VOL. LIV, NO. 2, FEBRUARY, 1959

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The Journal
of the
AMERICAN LEATHER CHEMISTS ASSOCIATION

Vol. LIV FEBRUARY 1959 No. 2

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SUBSCRIPTION - - MEMBERS $12.00 A YEAR - - NONMEMBERS $15.00

Published Monthly by

The American Leather Chemists Association

Entered as Second-Class Matter at the Post Office, Easton, Pa.
Acceptance for mailing at special rate of postage as provided for in section 1103, Act of October 3, 1917, authorized July 16, 1918

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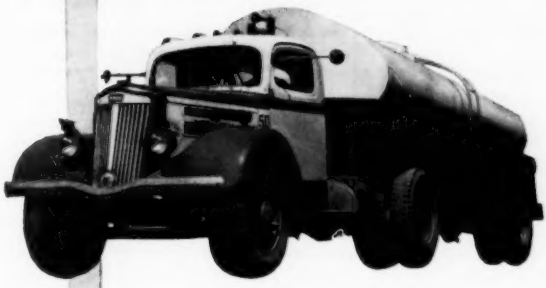

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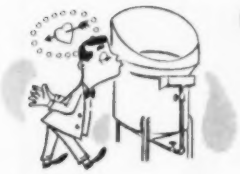

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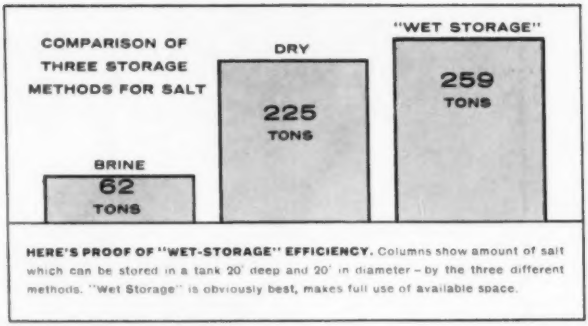

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General correspondence concerning the *Journal* should be mailed to the Managing Editor.

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STUDIES OF THE QUALITY OF LEATHER MADE FROM CALFSKINS OF CURRENT PACKING HOUSE PRACTICES*

ROBERT B. COMBS AND WILLIAM T. RODDY

Tanners' Council Research Laboratory
University of Cincinnati

and

JOSEPH NAGHSKI

*Eastern Regional Research Laboratory***
Philadelphia 18, *Pennsylvania*

ABSTRACT

Studies have been made to determine the quality of calfskin leather representative of two current packing house practices. In one system the skin remains on the carcass when it is placed in the cooler, to prevent shrinkage in weight of the carcass, and eventually the skin is removed in the cooler (cold pull). In the other system skinning is done immediately after slaughter (regular flay). Two lots of 120 calfskins, one of each practice, were analyzed and evaluated in the cure and as finished leather. No differences were found that could be attributed to the different practices or that indicated that one method was superior to the other as to leather quality. However, some interesting aspects of the relation of the strength of the raw stock to that of the finished leather have been noted, and such data are presented.

—×—×—

INTRODUCTION

The effect of current meat-handling practices on the quality of calfskin leather never has been established, although the skins from over ten million calves slaughtered annually in this country constitute an important by-

*A report of work done under contract with the U. S. Department of Agriculture and authorized by the Research and Marketing Act of 1946. The contract is being supervised by the Eastern Utilization Research and Development Division of the Agricultural Research Service. Presented at the Fifty-fourth Annual Meeting, Swampscott, Massachusetts, May 25, 1958.

**A laboratory of the Eastern Utilization Research and Development Division, Agricultural Research Service, United States Department of Agriculture.

Material supplementary to this article, consisting of tables giving analytical and physical test data on the individual skins, has been deposited as Document No. 5805 with the ADI Auxiliary Project, Photoduplication Service, Library of Congress, Washington 25, D.C. A copy may be secured by citing the document number, and by remitting $7.50 for photoprints, or $2.75 for a 35-mm. film. Advance payment is required. Make checks or money orders payable to Chief, Photoduplication Service, Library of Congress.

product having a total value of forty to fifty million dollars. There exist at present two major systems of handling calf carcasses. In one system the skin is left on the carcass to protect the surface of the meat and to prevent shrinkage in weight, a system similar to that used for handling hog carcasses. The other system is one in which skinning is done at the time of slaughtering.

The current use of both systems is permitted under federal and state meat inspection practices. If any marked advantage in values of the skins could be ascribed to either system of handling the carcasses, the industry would be able to use the preferred system.

Experimental study of the skins and leather produced from the two systems of handling the skins would establish the relative merits of the two systems. Because such research required both accurate and extensive knowledge of the art and science of handling the skins and processing them into leather, the United States Department of Agriculture entered into a contract with the Department of Tanning Research of the University of Cincinnati to perform the experimental work and set up the required tannery processing of the calfskins.

PLAN OF EXPERIMENTS

Through the cooperation of a hide broker it was possible to obtain cold-pull skins (skins removed from the carcasses in the packing plant cold room) and hot-flay skins (skins removed at time of slaughter) which were salted down in the broker's hide cellar on the same day, or on successive days, to make a lot. Four lots of 60 skins each were purchased and processed at two tanneries as indicative of cure cellar and tannery conditions during the summer and winter months.

One lot of 60 skins representing 30 cold-pull skins and 30 hot-flay skins was examined at the hide cellar for characteristics of the cure in November, 1955, and a 6″ x 8″ piece was removed from the kidney area of each skin (*see* Fig. 1) for histological assay, chemical analysis, and physical tests. A second lot and a third lot were examined and sampled during August, 1956. The fourth lot was examined and sampled in February, 1957. The extended period was necessary to permit the extensive testing and analysis required to evaluate the skins and the leathers produced.

After the skins of each lot were examined and sampled, they were shipped to tanneries where they were numbered and trimmed prior to processing as is also shown in Fig. 1. At Tannery 1 the skins were sorted as light- and heavy-weight skins and processed with the regular stock of the same weight. At Tannery 2, although no sorting was done, the skins were processed with the regular stock through chrome tannage and were finished as a tan aniline.

After being processed into leather at the two tanneries, the skins were graded for temper, grain break, veins, grain characteristics, fat wrinkles, and selling grade. The leathers were graded twice by two graders. The

second grading was done at a later date than the first and independent of the record of the first grading to eliminate bias. The skins were shipped to the Tanners' Council Research Laboratory for further testing. At the laboratory, a 6″ x 8″ piece was removed from the opposite side of each skin in a position corresponding to the one taken in the salt-cured condition. A histological assay, a chemical analysis, and physical tests were conducted on each leather piece.

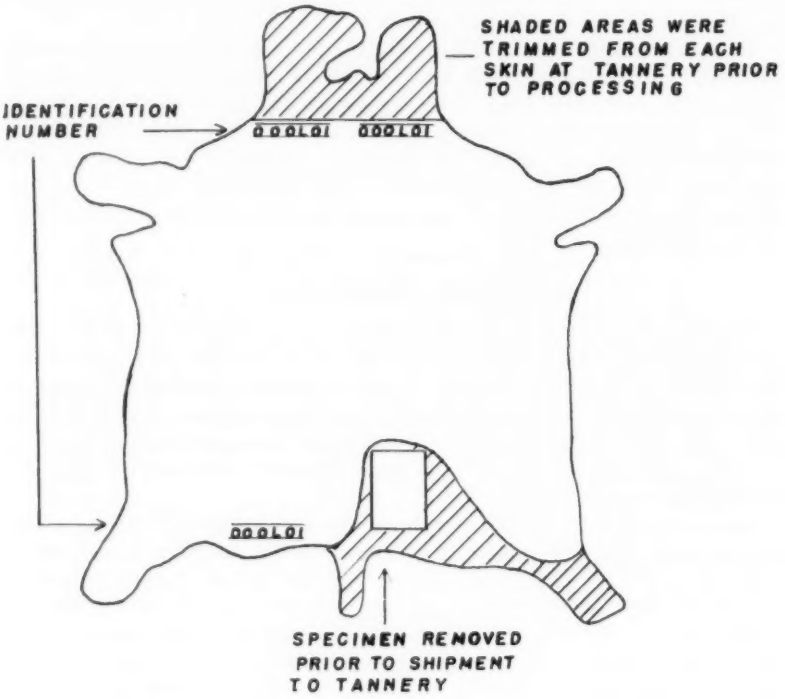
SHADED AREAS WERE TRIMMED FROM EACH SKIN AT TANNERY PRIOR TO PROCESSING
IDENTIFICATION NUMBER
SPECIMEN REMOVED PRIOR TO SHIPMENT TO TANNERY

FIGURE 1

CHARACTERISTICS OF THE SALT-CURED SKINS

For the purpose of evaluating the salt-cured skins, visual inspection, microscopic examination of cross sections, chemical analysis, and determination of tensile strength and stress-strain characteristics of each skin were conducted. By so doing it was possible to determine what differences, if any, occurred in the stock as a result of the packing house practices prior to shipping it to the tanneries for processing.

Visual inspection.—Each skin was inspected visually when it was spread across a sampling table at the broker's hide cellar. With the exception of the first 10 cold-pull skins, which showed some red heat, there was no condition apparent to the eye, nor any odor associated with the skins, that indicated poor cure. The skins were inspected again at the tanneries before processing. It was observed that the winter-cured skins showed no adverse effects as a result of shipment but that some of the summer-cured skins had dried areas when received at both tanneries.

Microstructure.—Cross sections were made of each salt-cured skin and stained for general structure and for fat in the epidermal area. A histological assay was obtained on each cross section by examination for epidermis slippage, fibroblasts in the corium, angle of the hair pocket, and deposition of fat in the epidermal area. Only 21 skins of the 240 calfskins examined showed epidermis slippage; in most of the skins the fibroblasts were still present; and the angle of the hair pocket varied in each lot—the drier the skin, the lower the angle. The deposition of the fat in the epidermal area was limited to the oil glands or their immediate vicinity and was similar for all skins, the only difference being that the larger skins contained more fat than the smaller ones. The characteristics examined for each lot were given numerical values, the total of the values constituting the histological assay of the lot. From the assays it was found that there were no significant differences among the four lots. Although microscopic examination of the 240 calfskins pointed out some imperfections, in general the individual lots of calfskins were very similar. None of the four lots showed any tendency toward alteration of the collagen fiber structure, and in most cases even the cellular structures of the skins were not unduly changed. Based on the histological assay of the salt-cured skins at the time they were ready for shipment to the tannery, it is indicated that there is no great difference in them as a result of the packing house practices of removing the skins.

Chemical analyses of salt-cured stock.—A portion of each 6″ x 8″ piece cut from the salt-cured skins was cut into small cubes for determining moisture, fat, protein, and total ash. An indication of quality of cure was secured through the determination of volatile nitrogen and total nitrogen. In addition each sample was analyzed for hydroxyproline as a measure of the percent collagen present.

The determinations of moisture, fat, protein, and total ash were conducted to secure a material balance of the composition of each skin. The cure "index" (3) was determined to ascertain the condition of each skin—stock with a cure index under 0.5 being a very acceptable cure. The hydroxyproline was determined in each case to obtain the percent collagen, because collagen is the only skin protein present which contains hydroxyproline.

TABLE I

CHEMICAL ANALYSES OF SALT-CURED SKINS

(Ranges and average values for 30 calfskins)

WINTER STOCK

Tannery 1

		Regular-Flay			Cold-Pull		
		Range		Average	Range		Average
Moisture	%	34.42	47.08	44.98	34.27	45.52	41.76
Fat	%	1.28	2.71	1.93	1.03	2.83	1.80
Protein	%	32.13	39.46	35.22	33.31	42.86	37.25
Total ash	%	15.25	22.54	17.34	14.48	22.92	17.19
Cure index		0.22	0.49	0.32	0.12	0.50	0.28
Collagen of protein	%	72.4	83.3	77.6	68.3	81.2	74.3

Tannery 2

Moisture	%	23.66	45.09	41.83	23.50	45.38	40.67
Fat	%	1.35	4.07	2.74	1.85	4.53	2.56
Protein	%	34.17	53.62	38.21	34.90	55.02	40.33
Total ash	%	15.54	20.46	17.03	11.23	22.77	15.95
Cure index		0.21	0.41	0.34	0.26	0.38	0.32
Collagen of protein	%	67.2	82.2	74.7	66.1	82.7	74.1

SUMMER STOCK

Tannery 1

Moisture	%	42.58	48.69	46.09	40.50	46.37	44.78
Fat	%	1.57	5.22	2.80	1.27	4.55	2.59
Protein	%	33.66	42.82	36.20	35.57	42.43	38.32
Total ash	%	9.66	15.89	14.20	12.21	15.01	14.02
Cure index		0.06	0.37	0.18	0.19	0.49	0.34
Collagen of protein	%	68.6	86.3	77.5	69.0	86.9	76.0

Tannery 2

Moisture	%	41.62	47.15	45.42	40.79	48.12	44.82
Fat	%	1.19	4.80	2.67	1.29	4.04	2.08
Protein	%	33.05	42.21	37.34	36.08	41.93	39.21
Total ash	%	12.24	15.26	14.12	12.10	15.53	13.81
Cure index		0.11	0.35	0.24	0.25	0.46	0.37
Collagen of protein	%	68.7	88.0	75.6	66.1	81.9	74.5

The chemical analyses of the calfskins are given in Table I. Each average value in the table represents 30 individual determinations; the ranges show the spread from the average. Examination of the data for moisture, fat, protein, and total ash shows that there is variation from skin to skin as to composition from the standpoint of all components. This variation in chemical composition occurs for all four lots of skins. While there is variation in chemical composition within a lot, when the range of values and average values are examined, it is seen that there is little difference in the summer- or winter-cured skins. The cure index indicates that all four lots of calfskins were well cured, and the percentage of collagen is similar for all four lots. The summer-cured skins have a higher average moisture content and a lower salt content, with the average protein content and fat content being the same for the summer- and winter-cured stocks.

Examination of the average chemical composition of the regular-flay stock versus the cold-pull stock indicates that there are no material differences in the two stocks. When the average value and range are compared, it can be seen that the stocks are similar as to moisture content, fat, collagen, and amount of salt present.

Strength of salt-cured stock.—From the 6″ x 8″ piece cut from each salt-cured skin, specimens for tensile strength, stress-strain characteristics, and bursting strength were secured. The object of determining strength characteristics of the salt-cured stock was to discover any differences which may occur due to the two packing house practices. To find out whether the strength characteristics may be altered by subsequent processing, the physical strength characteristics of the finished leather also were determined.

Prior to determining tensile strength, the specimens were measured for thickness in thousandths of an inch using the Randall-Stickney gauge. The tensile strength and stress-strain specimens were run on the Instron Tester using a 10-inch pulling speed, a chart speed of 50 inches per minute for measuring stretch, a 2-inch span between jaws, and a 500-pound full load. The stretch as well as the breaking load was recorded on the chart in each case.

The tensile strength and percent stretch values of the calfskins are given in Table II. Each average value in the table represents 30 individual determinations, and the ranges in each case show the spread from the average. Examination of the average tensile strength values in pounds per square inch for the regular flayed stock shows values of 3835, 2828, 3780, and 3418 lb. per sq. in. The values for the cold-pull stock were 3608, 2879, 2940, and 3078 lb. There is little difference in the tensile strength of the winter stock sent to Tannery 1, the average values being 3835 lb. for the regular flayed stock and 3608 lb. for the cold-pull stock. There is little difference in the tensile strength of the winter stock shipped to Tannery 2, the average values

TABLE II

TENSILE STRENGTH AND PERCENT STRETCH OF SALT-CURED SKINS

(Ranges and average values for 30 calfskins)

	Regular-Flay			Cold-Pull		
	Range		Average	Range		Average
WINTER STOCK						
Tannery 1						
Lb. at break	112	230	165	95	215	153
Thickness (inch)	.061	.111	.087	.060	.128	.090
% stretch	44.0	76.0	58.7	35.0	68.0	53.2
Lb. per sq. in.	2760	5410	3835	2650	5330	3608
Tannery 2						
Lb. at break	67	168	124	67	195	135
Thickness (inch)	.050	.122	.090	.062	.127	.094
% stretch	41.0	76.0	60.0	42.0	88.0	65.0
Lb. per sq. in.	1810	3500	2828	1440	3910	2879
SUMMER STOCK						
Tannery 1						
Lb. at break	130	265	198	105	225	150
Thickness (inch)	.078	.131	.105	.070	.146	.102
% stretch	37.0	81.0	61.0	38.0	75.0	57.0
Lb. per sq. in.	2310	4950	3780	2190	4270	3010
Tannery 2						
Lb. at break	145	230	188	98	201	153
Thickness (inch)	.084	.145	.111	.080	.155	.101
% stretch	58.0	91.0	71.0	45.0	98.0	63.7
Lb. per sq. in.	2410	4820	3418	2250	4270	3078

being 2828 lb. for the regular flayed stock and 2879 lb. for the cold-pull stock, but these values indicate lower average strength for the stock received at Tannery 2.

In the case of the summer-cured stock, the average tensile strength of the cold-pull stock (3010 and 3078 lb. per sq. in.) is lower than that of the regular flayed stock, which had average values of 3780 and 3418 lb. No evidence is available from the work performed as to whether the differences are due to

TABLE III

STRESS-STRAIN CHARACTERISTICS OF SALT-CURED SKINS

(Range and average values for 30 calfskins stretched 37.5%, relaxed, and then stretched to break)

	Regular-Flay			Cold-Pull		
	Range		Average	Range		Average
WINTER STOCK						
Tannery 1						
Lb. at break	82	200	156	95	195	147
Lb. at 37.5%	40	140	82	55	117	81
% stretch	42	85	62	45	75	59
Lb. per sq. in.	2500	4610	3434	2450	5060	3345
Tannery 2						
Lb. at break	85	195	139	66	210	130
Lb. at 37.5%	43	110	64	44	98	68
% stretch	46	86	68	51	80	66
Lb. per sq. in.	2160	5060	3075	2160	3800	2770
SUMMER STOCK						
Tannery 1						
Lb. at break	120	230	189	85	200	151
Lb. at 37.5%	60	150	115	45	140	89
% stretch	44	90	62	45	83	62
Lb. per sq. in.	2660	4630	3407	1900	4240	2970
Tannery 2						
Lb. at break	140	250	180	120	190	148
Lb. at 37.5%	35	110	77	35	110	75
% stretch	60	95	72	49	94	65
Lb. per sq. in.	2380	4310	3198	2240	3840	2959

the two packing house practices or to the handling of the skins after being removed from the carcasses, or whether they are characteristic of the animals from which the skins were taken.

The stress-strain test results of the winter and summer stocks are given in Table III. By extending the specimens to 37.5% elongation, allowing them to relax, and then extending them to break, there is a tendency to lower the average tensile strength values somewhat in all but the 30 skins of the regular-flay stock sent to Tannery 2. In comparing the values in Tables

II and III the regular-flay stock in Table II has average tensile strength values of 3835, 2828, 3780, and 3418 lb. per sq. in. compared with the values in Table III (specimens after straining before break and then breaking) of 3434, 3075, 3407, and 3198 lb. The cold-pull stock in Table II has average tensile strength values of 3608, 2879, 3010, and 3078 lb. per sq. in. compared to the values in Table III of 3345, 2770, 2970, and 2959 lb. Apparently when the salt-cured stock has a value of approximately 3000 or lower, the straining of the specimen to a constant extension of 37.5% and allowing it to relax before breaking does not alter the stock enough to prevent its recovery, whereas the specimens with the higher strength are definitely weakened, which results in lower tensile strength values upon applying strain before extending to break.

TANNERY EVALUATION

At each tannery the leathers made from the skins were graded for temper, grain break, veins, and grain characteristics, as well as for selling grade. At Tannery 1 the grading values obtained by two graders were averaged and submitted on this basis. At Tannery 2 the individual gradings made were submitted as such. The selling-grade values did not depend necessarily on the above determined characteristics but were very much dependent upon the useable area of the leather. Examination of the values given to the characteristics of the leather and to the selling-grade values indicated that there were no noticeable differences among the four lots of leather or between the leathers from the summer and winter stocks, based on the observations of the four graders at the two tanneries.

Microstructure of the leathers.—Cross sections were made of the leather from each of the 120 summer-cured skins processed at the two tanneries. The cross sections were measured for the curvature of grain side, hair pocket angle, and dye penetration.

The 60 calfskin leathers prepared at Tannery 1 showed little difference in the curvature of the grain and the hair pocket angle when the leather from the regular-flay versus the leather from the cold-pull calfskins were compared. The leather of the cold-pull calfskins showed greater dye penetration. There was little difference in the curvature of the grain, the hair pocket angle, or dye penetration of the regular-flay versus the cold-pull leather specimens from Tannery 2. The values obtained in the assay showed all the leathers from both tanneries to have similar characteristics, with only the dye penetration of the cold-pull stock from Tannery 1 being somewhat greater than observed in the other leathers.

Chemical analyses of leathers.—A portion of each 6″x 8″ piece taken from each skin was cut into cubes and ground in a Wiley mill to pass through a 4-mm. screen. This ground leather sample was used to determine moisture,

grease, hide substance, chromic oxide, and pH. The chromic oxide content was multiplied by 2.73, the "Kubelka factor" (1, 2), to calculate the content of chrome tanning agent. The chemical analyses of the leathers are given in Table IV. Examination of the data for grease, hide substance, and chrome tanning agent shows that there is variation in each lot as to composition but that the range of values is similar. Comparison of the data in Table IV indi-

TABLE IV

CHEMICAL ANALYSES OF LEATHERS

(Range and average values for 30 calfskins)

	Regular-Flay			Cold-Pull		
	Range		Average	Range		Average
WINTER STOCK						
Tannery 1						
Grease %	3.17	4.80	3.90	2.83	5.87	4.24
Hide substance %	81.11	84.48	82.60	78.45	85.36	82.28
Chrome tannage %	11.08	15.01	13.34	10.54	15.12	13.21
pH	3.62	4.73	3.76	3.51	3.90	3.71
Tannery 2						
Grease %	3.31	5.12	4.08	3.09	4.92	4.02
Hide substance %	80.04	84.57	82.78	81.23	86.09	82.99
Chrome tannage %	11.08	12.69	12.15	10.67	12.53	12.09
pH	3.03	3.53	3.32	3.23	3.42	3.30
SUMMER STOCK						
Tannery 1						
Grease %	3.45	5.57	4.45	3.51	6.36	4.45
Hide substance %	80.96	86.09	82.49	77.73	84.81	82.23
Chrome tannage %	11.14	12.53	12.09	12.18	14.96	13.46
pH	3.22	3.40	3.30	3.47	4.03	3.59
Tannery 2						
Grease %	3.04	6.03	4.54	3.24	6.72	3.95
Hide substance %	79.33	86.62	82.80	82.23	85.69	83.96
Chrome tannage %	12.07	14.58	13.47	10.54	11.85	11.36
pH	3.50	3.86	3.59	3.35	3.48	3.42

TABLE V

TENSILE STRENGTH AND PERCENT STRETCH OF LEATHER

(Range and average for 30 calfskins)

	Regular-Flay			Cold-Pull		
	Range		Average	Range		Average
WINTER STOCK						
Tannery 1						
Lb. at break	55	133	93	70	125	99
Thickness (inch)	.039	.059	.050	.028	.074	.049
% stretch	25	57	39	26	58	36
Lb. per sq. in.	2620	5130	3755	2190	6640	4091
Tannery 2						
Lb. at break	43	97	62	36	97	67
Thickness (inch)	.027	.053	.041	.028	.057	.042
% stretch	27	58	39	26	52	39
Lb. per sq. in.	2300	4510	3048	2440	4220	3176
SUMMER STOCK						
Tannery 1						
Lb. at break	40	115	79	40	105	74
Thickness (inch)	.032	.065	.051	.033	.066	.048
% stretch	30	60	45	33	82	47
Lb. per sq. in.	2100	4370	3111	2240	3780	3120
Tannery 2						
Lb. at break	42	120	76	50	98	75
Thickness (inch)	.041	.068	.053	.042	.067	.052
% stretch	32	60	47	35	76	51
Lb. per sq. in.	1820	4000	2868	1850	4090	2943

cates that there is practically no difference in the average chemical composition of the regular-flay versus the cold-pull calfskin leathers. There is variation in acidity of the leathers, but this represents plant practices.

Strength of the leathers.—From each 6″ x 8″ piece taken from each skin, specimens were cut for determining tensile strength, percent stretch at break, and stress-strain characteristics, by the procedure used for the salt-cured skin specimens. The tensile strength and percent stretch at break for the 240 leathers are given as average and range values in Table V. Examina-

TABLE VI

STRESS-STRAIN CHARACTERISTICS OF LEATHER

(Range and average values for 30 calf skins stretched 37.5%, relaxed, and then stretched to break)

	Regular-Flay			Cold-Pull		
	Range		Average	Range		Average
WINTER STOCK						
Tannery 1						
Lb. at break	45	120	96	60	130	101
Lb. at 37.5%	58	107	80	55	130	87
% stretch	35	63	43	30	53	41
Lb. per sq. in.	2500	4880	3931	3110	6000	4330
Tannery 2						
Lb. at break	49	93	64	47	94	66
Lb. at 37.5%	36	66	53	39	79	52
% stretch	30	56	43	30	61	44
Lb. per sq. in.	2270	4280	3135	2170	4370	3131
SUMMER STOCK						
Tannery 1						
Lb. at break	38	110	80	43	105	75
Lb. at 37.5%	25	81	59	30	87	60
% stretch	34	66	50	33	74	49
Lb. per sq. in.	2110	4250	3109	2030	4130	3121
Tannery 2						
Lb. at break	43	102	70	40	110	74
Lb. at 37.5%	25	90	52	30	80	65
% stretch	36	99	51	32	70	46
Lb. per sq. in.	1870	4070	2653	1630	4290	2832

tion of the data in Table V shows that the winter stock processed at Tannery 1 has higher average tensile strength for both regular-flay and cold-pull calfskin leathers than that obtained for the other six groups given. This is a reflection of the original tensile strength of the salt-cured stock from which the leathers were made as given in Table II. The other six groups which have lower average tensile strength as leather also reflect the lower tensile strength of the salt-cured stock as given in Table II. On the basis of these

results there was no difference in strength properties brought about by tannery practices.

The stress-strain characteristics of the regular-flay and cold-pull calfskin leather specimens processed at the two tanneries are given in Table VI. Upon extending the specimens to 37.5% (which was the extension selected for the salt-cured calfskins), allowing them to relax, and then extending them to break, there was no tendency toward lowering of the tensile strength as observed in the case of the salt-cured skins. In comparing the values in Tables V and VI the regular-flay stock in Table V has average tensile strength values of 3755, 3048, 3111, and 2868 lb. per sq. in. compared to the values in Table VI (specimens after strain before break and then breaking) of 3931, 3135, 3109, and 2653 lb. The cold-pull stock in Table V has average tensile strength values of 4091, 3176, 3120, and 2943 lb. per sq. in. compared to the values in Table VI of 4330, 3131, 3121, and 2832 lb. When the values for percent stretch at break are compared, it is observed that the summer stock

TABLE VII

COMPARISON OF THE STRENGTH IN POUNDS OF REGULAR-FLAY AND COLD-PULL CALFSKINS AND LEATHERS

Regular-Flay		Cold-Pull	
WINTER STOCK			
Tannery 1			
Skin	165	Skin	153
Leather	93	Leather	99
Tannery 2			
Skin	129	Skin	135
Leather	62	Leather	67
SUMMER STOCK			
Tannery 1			
Skin	198	Skin	150
Leather	79	Leather	74
Tannery 2			
Skin	188	Skin	150
Leather	76	Leather	74

processed at both tanneries has a greater stretch than the winter stock, which might reflect a slightly greater process action on the stock during summer tannery processing.

COMPARISON OF SALT-CURED SKINS AND LEATHER

The tensile strength on a pounds-at-break basis and as pounds per square inch of the skins and leathers is given in Tables VII and VIII. A comparison of the regular-flay and cold-pull skins and leathers in Table VII shows that there is a very noticeable difference in the strength of skins versus the leathers. The leathers have a much lower breaking strength on the basis of pounds at break. However, during processing, the skins were shaved to a standard thickness which results in decreasing their strength because there is less fiber structure to be broken. When this thickness reduction is taken into account, the values in Table VIII show that there is little variation in the strength measurements for salt-cured skin and leather.

TABLE VIII

COMPARISON OF THE STRENGTH IN POUNDS PER SQUARE INCH OF REGULAR-FLAY AND COLD-PULL CALFSKINS AND LEATHERS

Regular-Flay		Cold-Pull	
WINTER STOCK			
Tannery 1			
Skin	3835	Skin	3608
Leather	3755	Leather	4091
Tannery 2			
Skin	2828	Skin	2879
Leather	3048	Leather	3176
SUMMER STOCK			
Tannery 1			
Skin	3780	Skin	2940
Leather	3111	Leather	3120
Tannery 2			
Skin	3418	Skin	3078
Leather	2868	Leather	2943

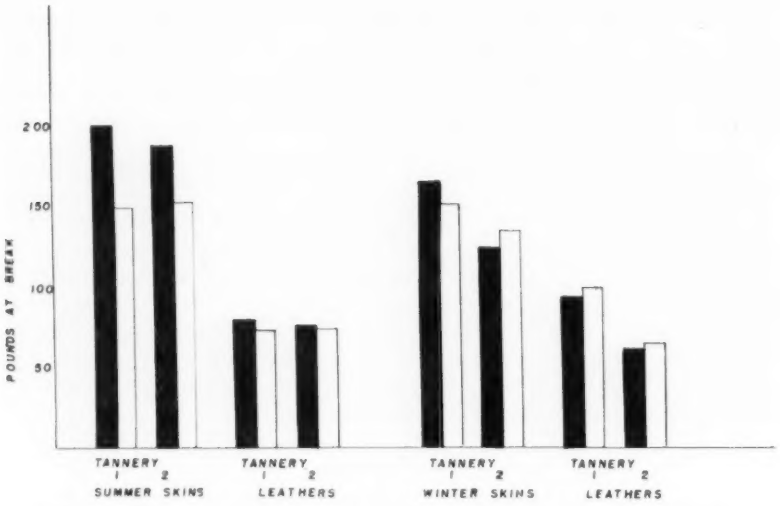

FIGURE 2.—Tensile strength. Pounds at break. Regular flay in black; cold pull in white.

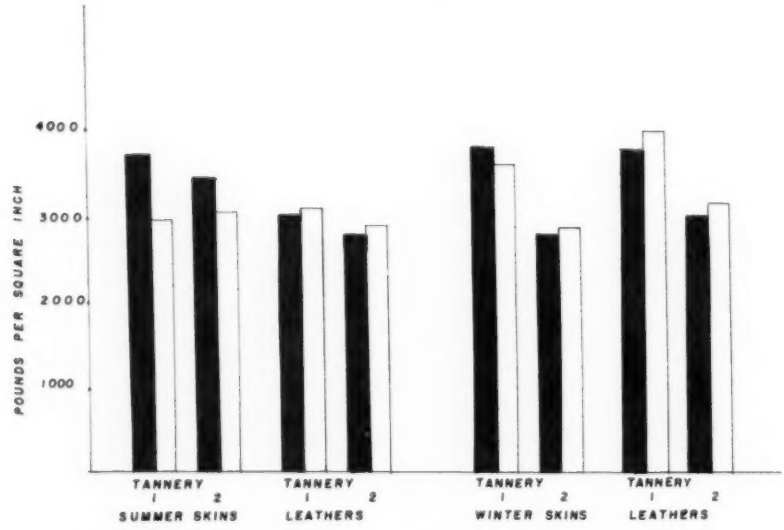

FIGURE 3.—Tensile strength. Pounds per square inch at break. Regular flay in black; cold pull in white.

Examination of the data in Table VIII shows that the winter skins processed at Tannery 1 have comparable tensile strengths as leathers irrespective of whether they were hot-flay or cold-pull skins. The winter skins processed at Tannery 2 have lower initial strength irrespective of take-off practices than those sent to Tannery 1, but they have comparable tensile strength as leather.

The regular-flay summer skins processed at both tanneries show a reduction in tensile strength when made into leather. Seemingly, this reduction may be the result of processing at both tanneries, but just what it reflects is not known from our present experimental plan. The reduction in thickness due to processing is not the only factor involved in the reduction of strength in this case. The cold-pull summer skins processed at each tannery do not show a reduction in strength as a result of processing but had lower strength than the regular-flay skins before being received at the tanneries.

The data in Tables VII and VIII are given in bar-graph form in Figs. 2 and 3. These figures bear out the observations mentioned above and also show that the summer stock of the regular-flay calfskins requires a greater number of pounds to break it than is necessary for the cold-pull calfskins. This difference is not observed in the leathers. Although the reduction of tensile strength of the regular-flay stock is readily observed, in the case in the winter skins, processing into leather has not decreased the tensile strength.

From the elongation curves of the leather specimens used for tensile-strength measurements, the average values of load at 10, 20, and 30% extensions were used to give the curves shown in Figs. 4 and 5. In Fig. 4 the curves for the stock processed at Tannery 1 are given. When the regular-flay versus the cold-pull calfskin leather specimens are compared, it is observed that the summer stock is more readily extended than the winter stock, requires less load in pounds to extend the leathers, and breaks at a greater extension but at lower loads.

The curves for the stock processed at Tannery 2 are given in Fig. 5. These leathers have similar properties for both summer and winter cure and are similar to the summer-cured stock processed at Tannery 1. The winter stock processed at Tannery 1 is less extensible than the other six lots.

From the stress-strain curves, where the stock was extended first to 37.5%, allowed to relax, and then stretched to 40% again, the curves in Figs. 6 and 7 were plotted. These curves reflect the permanent set put into the stock as a result of the first extension prior to extending the specimens again, and it is observed that the leather is more stretchy on the second extension at the lower loads.

The curves shown in Fig. 6 represent the summer and winter cure and processed stock at Tannery 1. The summer stock is more readily extended than the winter stock. The curves shown in Fig. 7 represent the summer and winter cure and processed stock at Tannery 2. This stock gives curves that are similar to the summer stock processed at Tannery 1.

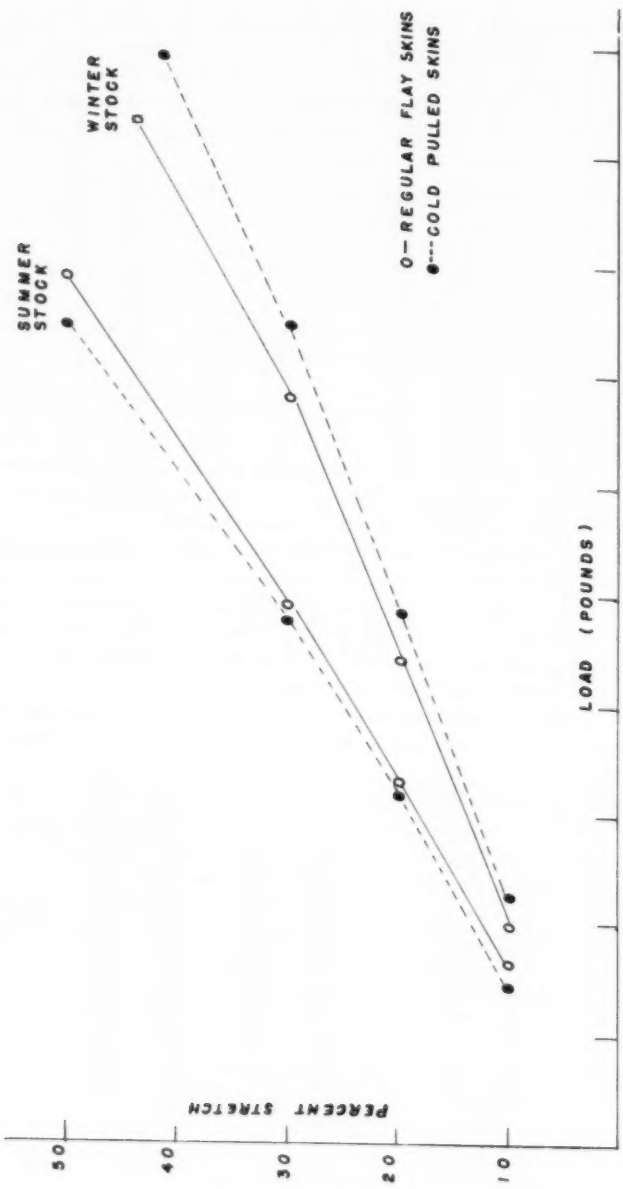

FIGURE 4.—Load versus stretch of tensile curves of Tannery 1. Summer and winter cure and process.

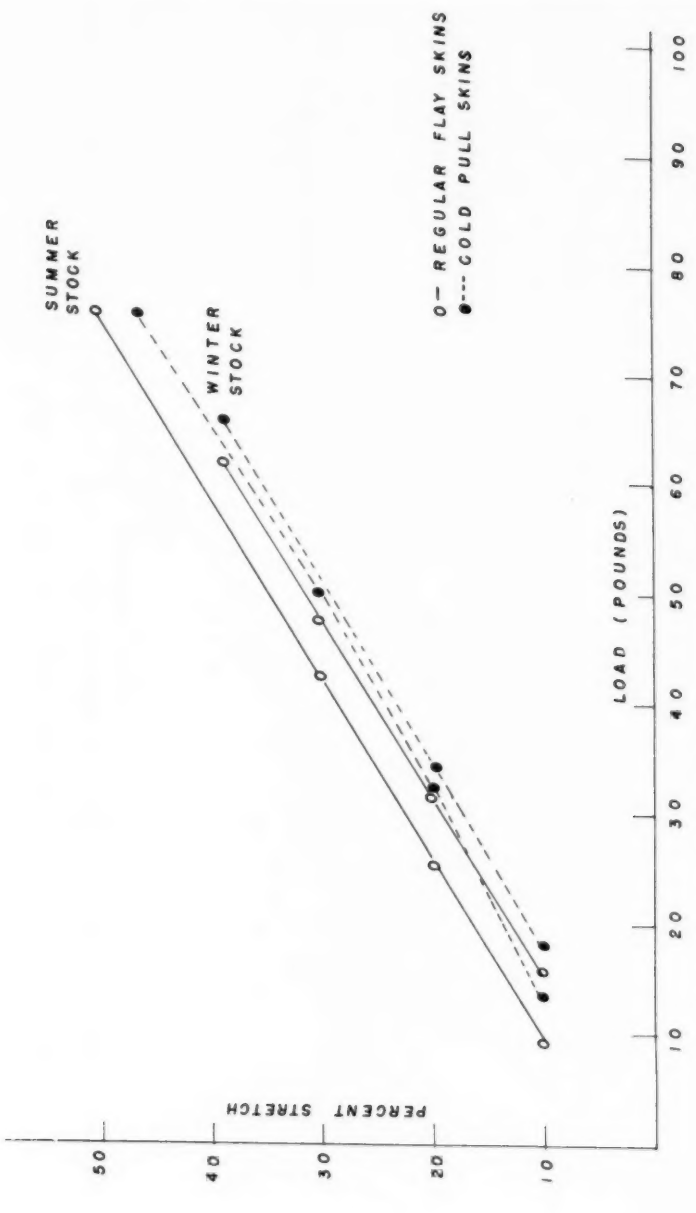

FIGURE 5.—Load versus stretch of tensile curves of Tannery 2. Summer and winter cure and process.

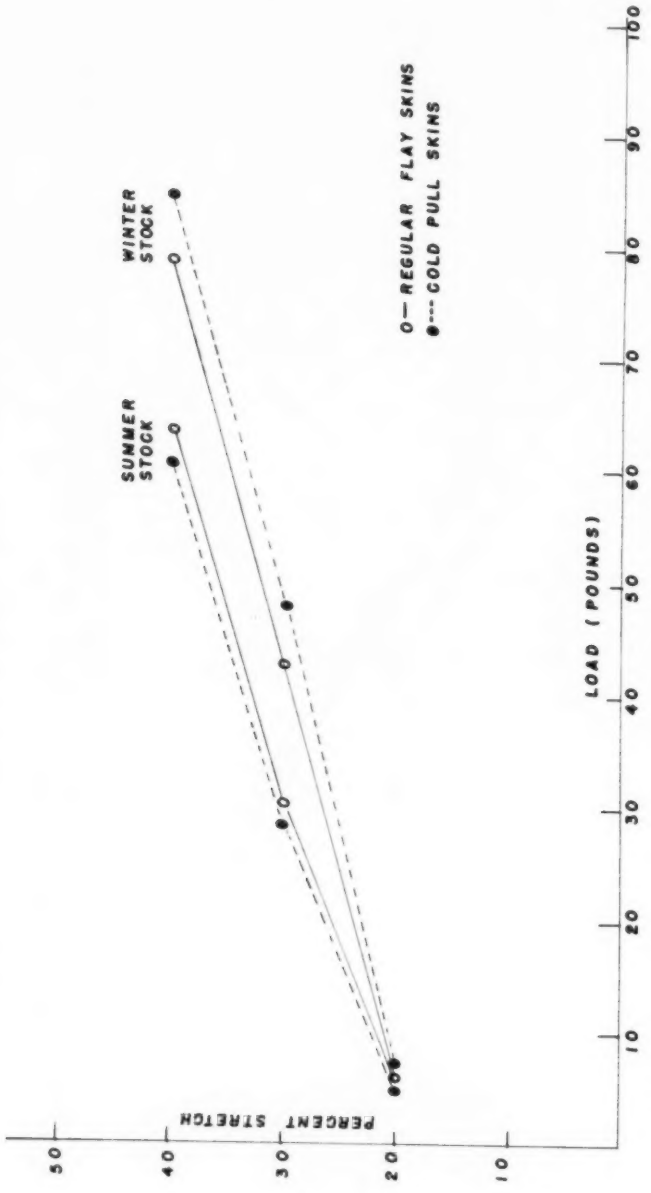

FIGURE 6.—Load versus stretch after elongation to 37.5%, relaxation 1 minute, and stretch to 40%. Tannery 1.

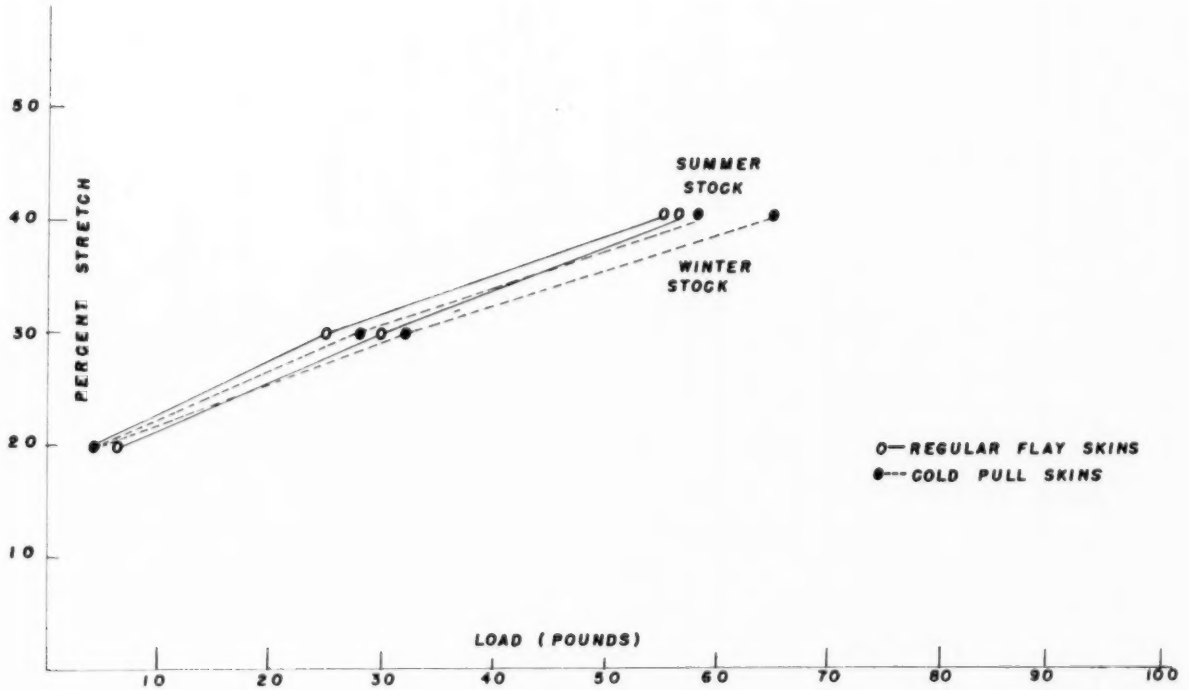

FIGURE 7.—Load versus stretch after elongation to 37.5%, relaxation 1 minute, and stretch to 40%. Tannery 2.

CONCLUSIONS

Based on visual inspection, microstructure, chemical analyses, and strength of the salt-cured stock, there were no detectable differences in the cold-pull skins versus the regular-flay skins. When the skins were processed into leather at the two tanneries and examined in a manner similar to that used in assaying the salt-cured stock, no differences could be found due to the practices of removing the skins from the carcasses, nor was there any indication that one method was superior to the other relative to leather quality.

The tensile strength of the salt-cured stock was not noticeably altered by processing at either tannery. The leathers made from the salt-cured skins had approximately the same tensile strength in lb. per square inch as the salt-cured skins. Less force was required to break the leathers than the salt-cured skins because of the reduction in thickness to bring the leather to a more uniform thickness. Processing in both tanneries was such that the strength of the salt-cured stock was retained or slightly reduced in one case but not improved.

The only difference found among the leathers was in the extensibility of the winter stock at Tannery 1. The stock was firmer than the other three batches of skins processed and probably reflected the manner in which the calfskins were handled in the tannery.

ACKNOWLEDGMENTS

In obtaining the data presented in this paper the cooperation and the assistance of the staff of the Tanners' Council Research Laboratory, particularly V. Mattei, R. Meier, D. Moser, J. Tancous, and K. Veith, have been very valuable.

Use of the facilities of the B. N. Ritter Company, Cincinnati, Ohio; the Clarence Rice Packing Company, Covington, Kentucky; the A. C. Lawrence Leather Company, Peabody, Massachusetts; and the Ohio Leather Company, Girard, Ohio, is gratefully acknowledged. Special appreciation is extended to Messrs. B. Ritter, J. Sway, M. Battles, and C. Ross.

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Received August 13, 1958.

DISCUSSION

HAROLD R. MILLER, JR. (E. I. duPont de Nemours & Co.): In leather research as well as in other forms of research, a definite result is all-important,

whether it be positive or negative. In this case, your decision depends on which side of the fence you are on—whether you are a tanner or a packer. Although I am no longer in the tanning industry in a production capacity, my sentiments are still along that line. To me this looks like a negative result in that it tends to make me think that here is an opportunity to eliminate another condition prior to the actual tanning operation as affecting results.

I would like to ask Mr. Roddy whether he would like to elaborate on why the one lot of winter take-off skins was different from the other?

MR. RODDY: That gets us into the processing of the skins at the tanneries. As you can see, at tannery No. 2 the skins behaved the same; whereas, at tannery No. 1, during the winter months, the skins apparently were not let out as much as they were during the summer months, or as they were in both cases at tannery No. 2.

This is a reflection, apparently, of temperature conditions in tannery No. 2 during the winter months, and while the liquors—beamhouse and soak liquors—are kept at the same temperature during the winter months, they also try to approach the same temperature during the summer months.

At tannery No. 1, using the type of unhairing process they do, they get less action on the skin during the winter months than during the summer months, which apparently is reflected in the results obtained.

MR. MILLER: From the correlation of analyses between cured stock and finished leather, does there seem to be any hope of eventually setting up a system of inspection, a more foolproof method than just visual inspection, to predict leather quality from the cured condition?

MR. RODDY: During the past few years at the University of Cincinnati we have been analyzing the cured stock as well as finished leather, not only from the standpoint of chemical analysis but also the physical characteristics or strength measurements. And this entire paper indicates that strength measurements can be used as an evaluating tool for strength-stretch measurements.

C. DAVID WILSON (Rueping Leather Co.): Was each skin identified? Did each skin have its own identity?

MR. RODDY: Yes, each skin was identified.

MR. WILSON: What was the degree of correlation, then, skin for skin, between skin strength and leather strength? Or did you perform such a correlation or look for it?

MR. RODDY: We definitely looked for it. I would mention that even on the skins where the original thickness was retained in the finished leather, in some cases we came up with the same strength. In other cases there was reduction of strength.

MR. WILSON: Over all, though, in the lot of 240 skins, is the correlation between skin strength and leather strength a good correlation?

MR. RODDY: A fairly good correlation, yes.

DR. R. M. LOLLAR (Tanners' Council Research Laboratory): I would like to extend that correlation to hides, on which I have done more work. The correlation between hide strength and strength of leather made on the same hides is in general positive and statistically significant. But to a practical extent it is not as good as we would like.

DR. MELLON (Eastern Utilization Research Laboratory): Am I correct in my belief, from what you were saying, that the difference that you describe between the winter stock and summer stock is due to the time at which it was processed in the plant?

MR. RODDY: It represented differences both from the standpoint of the cure and from the standpoint of processing—either summer or winter cure, or summer or winter processing. The summer-cure skins were processed during the summer and the winter skins were processed during the winter.

DR. MELLON: The object of my question was to find out whether the difference was due to processing or whether it indicated a change in the animal. I cannot quite see a change in the animal's skin, whether it was taken off in the summer or winter, because the collagen structure is what you are studying in the leather, and it does not change very rapidly. There is not the rapid change-over of collagen as there is with muscular tissues.

MR. RODDY: I did not go into the chemical analysis this morning, but I can tell you that from the standpoint of chemical composition of both the skins and leathers we would not be able to draw the decisions we have drawn if we had not run the physical tests.

To Prepare Transparent Leather. The cleansed skin is repeatedly coated with a mixture of 100 parts of glycerine, 0.2 of salicylic acid, 0.2 of picric acid, and 2.5 of borax; then nearly dried and impregnated in a dark room with a solution of bichromate of potassium; then completely dried and coated on both sides with shellac varnish.
—*The Techno-Chemical Receipt Book.* Philadelphia: Henry Carey Baird & Co., 1903.

POLYSULFIDE RUBBER TREATMENT FOR GLOVE AND SHOE UPPER LEATHERS*

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QM Research and Engineering Center
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ABSTRACT

A process is described for treating cowhide glove and similar flexible types of leather with liquid polysulfide polymers to produce a high degree of resistance to penetration or absorption of petroleum products, liquid chemicals, and water. The polymer is applied to the grain layer only and is heat-cured in the usual drying tunnel to form a protective barrier in the leather without serious detraction from the feel or flexibility of the leather or the ability of the flesh side to absorb perspiration. A small-scale tannery trial is described showing the possibility of plant application without difficulty. The results of a field-wear trial of treated leather gloves are presented.

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INTRODUCTION

Satisfactory performance of leather in gloves, shoes, and other clothing items is dependent upon a combination of properties, including strength and abrasion resistance for durability, absorption and vapor permeability for comfort, and air space between fibers for insulation. These characteristics of leather in the normal dry state are generally satisfactory, but they may be adversely affected in leather that comes in contact with water or other liquids. The problem dates perhaps to antiquity, but only during the last few years has there been real progress toward a satisfactory solution. Several methods have been described for improving the resistance of leather to absorption or penetration of water (1–4), but much less attention has been given to other liquids.

The exposure of military leather items to gasoline, oil, and other petroleum products ("POL" products in quartermaster terminology) has presented a serious problem, particularly under extreme-cold conditions. For example, there have been instances in which Arctic deerskin mittens have become so saturated with POL products within a few weeks of wear that most of their insulating value was lost and they were a potential fire hazard, especially if in close proximity to an open fire.

*Presented at the Fifty-fourth Annual Meeting, Swampscott, Massachusetts., May 27, 1958.

This paper describes experiments designed to solve the problem by improving the resistance of leather to absorption and penetration of petroleum products, water, and other liquids. A tannery-trial application of an impregnation process is described, as well as the results of laboratory evaluations and a limited wear trial of treated cowhide leather gloves.

EXPERIMENTAL

Surface coatings.—In the exploratory studies for producing a POL-resistant glove leather, attempts were made to develop a treating material which could be easily applied to the grain side of leather to form a liquid-resistant, light but serviceable, plastic-type, surface coating. At the time, this approach appeared to offer the most promise as a solution of the problem, more so than any available impregnating process. It was erroneously assumed from earlier impregnation studies that the desired resistance to POL products could be attained only through the use of large quantities of impregnating materials which would impair leather characteristics such as flexibility.

Various materials were investigated as surface films on horsehide and deerskin glove leather, including acrylates, neoprenes, vinyl or vinylidene chlorides, and various polyethylenes. Laboratory tests showed that coatings less than 10 mils thick provided good resistance to gasoline for more than 10,000 cycles with the Maeser Tester and that the feel and softness of the leather were also acceptable. However, surface coatings of this type were too stiff for use at low temperatures and lacked durability. For example, in one wearing trial 10 pairs of leather gloves were coated to approximately 8 mils thickness with a promising polyvinylidene chloride and butadiene-acrylonitrile mixture and worn by workmen engaged in normal handling operations involving gasoline and other POL products. Within three days, scuffing and peeling of the gloves had become so bad that the gloves offered no better resistance than the untreated controls.

Impregnation.—The unsuccessful experiments with surface films led to a re-evaluation and study of impregnation processes and materials. Screening and application studies were performed on a number of potential impregnating agents such as epoxy resins, acrylates, liquid polysulfide polymers, polyethylenes, and special oils, greases, or waxes. Of the materials investigated, the ones that showed greatest promise were the liquid polysulfide polymers. These liquid, low-molecular-weight, polymercaptan polymers are capable of further polymerization into rubber-like elastomers with unusually high resistance to oils and solvents and with good aging and low temperature properties.

Although commercial methods for applying the liquid polysulfide polymers to leather have been described in this journal (5) and in technical bulletins

(6), their principal application apparently has been in packings, belting, and other mechanical leathers. These commercial methods use a process of total immersion and thorough impregnation of the leather. These processes proved to be unsatisfactory for glove or shoe upper leather, nor could any satisfactory method based on thorough impregnation be developed in the present work by which leather could be obtained with good liquid resistance and without loss of desired leather characteristics.

During some exploratory work on emulsions of the polysulfide polymers, in which morpholine was employed in the dual capacity of emulsifier and amine curing agent, it was found that these emulsions could be applied directly to the grain side of the leather by brushing or rolling to produce a leather of fair liquid resistance and good leather characteristics. Further work with solutions composed of the polysulfide polymer blended with morpholine as the curing agent and solvents such as toluene, perchloroethylene, and finally tetrahydrofurfuryl alcohol proved to be even more effective.

POLYSULFIDE POLYMER TREATMENT

In essence, the treatment consists of applying a polysulfide solution to the grain surface of the leather in a manner similar to hand-swabbing. Immediately following the application, the leather is heated at 140°–160°F. in a chamber or drying tunnel with good air circulation for 15–60 minutes and then placed in a dry loft at approximately 90°–100°F. for 16–24 hours. The treatment produces a permanently fixed, flexible, polymeric layer within the leather approximately 10–15 mils thick and extending from the grain surface inward. This treated leather is capable of effectively resisting penetration by liquids such as water, POL products, and certain corrosive chemical solutions. The flesh side of the leather remains unaffected by the treatment and, hence, retains its comfortable feel and perspiration-absorption properties. Furthermore, the treatment has only a slight effect upon strength or other leather characteristics.

The treating materials and some of their physical properties are shown in Table I.

The liquid polysulfide polymer Thiokol LP-32 was found to produce leather of good liquid resistance and best over-all leather characteristics compared to other liquid polysulfide polymers and blends. This product is supplied in the form of a viscous liquid which must be diluted with a solvent for satisfactory penetration into leather. Both morpholine and tetrahydrofurfuryl alcohol (THFA) serve in a dual capacity: morpholine as an amine curing agent, THFA as a copolymerizing agent, and both materials as diluents. Oleic acid, a retarding agent, has been found useful in many cases in facilitating penetration and improving the distribution of the polymer within the leather.

TABLE I

TREATING MATERIALS AND THEIR PHYSICAL CHARACTERISTICS

(Information from manufacturers' bulletins)

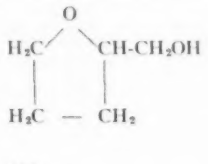
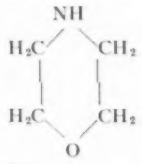
Physical Characteristics	Polymer Thiokol LP-32 (Thiokol Corp.)	Tetrahydrofurfuryl-alcohol (Quaker Oats Co.)	Morpholine
Structure	(HS-R-SH)*n*		
Approx. molecular weight	4,000	102	87
Specific gravity	1.27(20°/4°C.)	1.05(20°/20°C.)	1.00(20°/20°C.)
Vapor pressure, mm. Hg	Very low	2.3(41.6°C.)	8(20°C.)
Viscosity, poises	350-450(25°C.)	0.062(20°C.)	0.023(20°C.)
Flash point, °F.	455	183	100

TABLE II

TREATING SOLUTIONS

Materials (parts by weight)	Solution A	Solution B
Thiokol LP-32	100	100
Tetrahydrofurfuryl alcohol	45	75
Morpholine	10	
Oleic acid	0.5	
PHYSICAL PROPERTIES		
Specific gravity, 78°F.	1.15	1.16
Viscosity, centipoises, 78°F.	880	710
pH (water extract)	9.5	7.3
Flash point, open cup, °F.	170	169

Table II shows the composition of two treating solutions which have consistently produced good results, especially with cowhide glove or garment leathers.

In Solution A, morpholine, which was used in many of the earlier formulations on glove leather, is employed instead of some of the more reactive amine or paint drier-type curing agents, e.g., diethylamine or cobalt hexogen. Morpholine has several important advantages: not only is it sufficiently effective for this treatment but it is easily removed by heat during the curing process, causes no apparent damage to leather, and is available and already in use within the leather industry.

During our studies with THFA as a solvent it was found that this material is capable of copolymerizing with some of the liquid polysulfide polymers. The reaction may be similar to that described in the literature for furfural (6), although considerably slower in rate. Solution B contains only the polysulfide polymer and THFA. This solution has several advantages over Solution A, being simpler in composition, penetrating more quickly, and generally providing greater resistance in the treated leather. However, certain types of leather, containing resins or other material that apparently act as anticatalysts, require morpholine for an effective treatment. In these cases Solution A should be used.

The most convenient methods of application, on a laboratory scale, were by rolling with a paint roller, brushing, or swabbing pieces of leather approximately 1 square foot in size. Whole sides were easily treated in the same way. Usually the treated leather was placed within 5 minutes into a drying cabinet with rapid air circulation at 140°–160°F. for one hour (Solution A) or 15 minutes (Solution B). The leather was then maintained at 90°–100°F. overnight for further curing and removal of the residual solvents.

Preliminary application of the polymer to finished commercial garment leather was ineffective in areas containing a high concentration of oil. In order to eliminate this difficulty subsequent laboratory tests and tannery trials were made on degreased leather.

During the first tannery experiment 10 sides of cowhide glove leather were treated by swabbing with a solution containing 30 parts morpholine to 100 parts polysulfide polymer. Although room temperature on the day of the experiment was low, the highly viscous formulation penetrated readily in most places, imparting good resistance to penetration of water and POL products, but it failed to produce this effect uniformly over the entire area of the sides.

A second tannery trial was made on 15 sides of cowhide glove leather in order to evaluate a spray formulation consisting of polysulfide polymer, morpholine, and perchloroethylene. In this trial the liquid resistance of the leather was relatively low and very spotty over all of the sides. Weight measurements of the treated leather showed that material losses were approximately 50%, which shows why the poor results might have been expected.

A third tannery trial comprising 30 sides of degreased cowhide glove leather proved more successful than previous trials. Solution B of Table II was applied to the grain side of the leather, using a mohair paint roller to spread the solution quickly and uniformly. The amount of solution applied was calculated so as to impart 18 grams of polymer, or approximately one ounce of solution, per square foot of leather. After treatment the sides were hung in a drying tunnel at 140°–160°F. for 15 minutes and then overnight in a dry loft at approximately 90°F. The dried leather was washed for 20 minutes in a drum at 125°F., fatliquored with 4% oil, dried, and staked.

EVALUATION OF TREATED LEATHER

The effect of polysulfide polymer concentration on water resistance was determined on 5 sides of degreased cowhide glove leather. Single pieces from each side were treated to contain 9, 12, 15, 18, 21, and 24 grams polymer per square foot of leather. The leather was washed, fatliquored, dried, and staked after treatment. Resistance to water penetration was measured by the QM Tap Tester (7) using a ½-lb. load. Figure 1 shows the number of cycles required for initial water penetration.

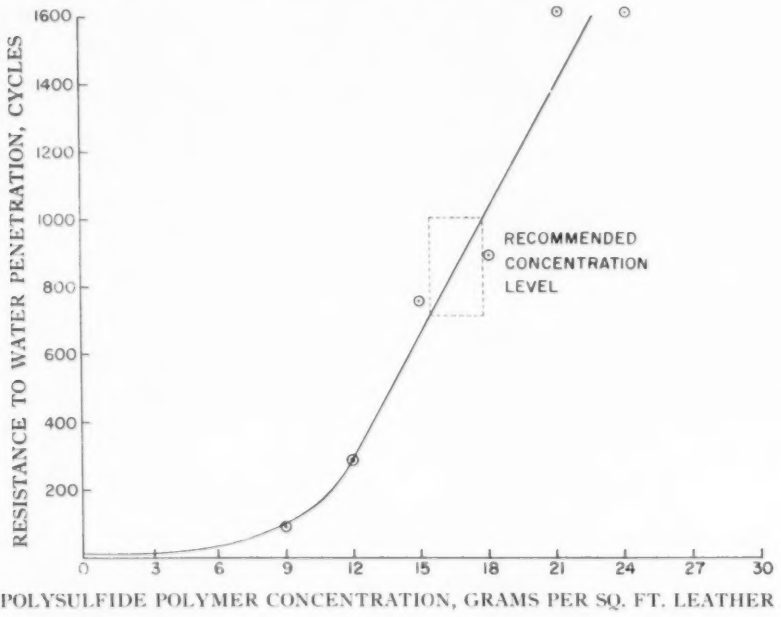


FIGURE 1.—Water resistance for cowhide glove leather at different polymer concentration.

Resistance to water penetration increases with increasing concentration of the polymer from approximately 20 cycles for untreated leather to 1600 cycles for leather treated at the 24-gram level. No other treatment except a Bavon process (8) provided a similar high resistance to water penetration. The increase in resistance is directly proportional to the polymer concentration within the range of 9 to 24 grams. Also indicated in Fig. 1 is the recommended concentration level for glove leather which is 15 to 18 grams polymer per square foot of leather. This suggested level was selected on the

basis of a combination of properties, including water- and POL-resistance and other characteristics related to comfort and performance in gloves. Higher treating levels provided greater water resistance but at a sacrifice in water vapor permeability and softness as well as adding to the cost and also to the weight of the gloves.

Because of the inherent difference in hide structure in different areas of a hide, some variation in properties is unavoidable. The effects of this variation were studied on ten of the sides treated in the third tannery trial previously described. Results of the tapping test for these leathers are presented in Table III.

TABLE III

VARIATION IN WATER RESISTANCE IN A LOT OF TANNERY-TREATED SIDES OF COWHIDE GLOVE LEATHER

Location on Side	Number of Measurements (2 for each area)			
	Below 100 Cycles	100-299 Cycles	300-599 Cycles	Above 600 Cycles
Backbone-tail	0	3	10	7
Backbone-center	1	4	5	10
Belly-center	2	4	4	10
Shoulder	2	10	3	5
Total	5	21	22	32
Side number				
1	4	3	1	0
2	0	5	1	2
3	1	2	3	2
4	0	3	4	1
5	0	2	5	1
6	0	2	2	4
7	0	1	3	4
8	0	2	1	5
9	0	1	2	5
10	0	0	0	8

The results in the upper part of the table show that 32 of the 90 measurements did not produce a leak in 600 cycles, thus indicating a relatively high degree of water resistance. Only 5 values fell below the 100-cycle level, the lowest value being 71 cycles. Resistance was generally least in the shoulder area as compared to the other areas tested. The table also shows the variation in results for treated sides in the same lot. The results were uniformly high on eight of the sides, whereas the other two showed a significant difference in water resistance. The poorest side was No. 1 with four values below 100 cycles compared to only one similar value for all other sides. The highest

degree of water resistance in the lot was that of side No. 10, which did not leak in any test.

Another method of measuring water resistance was used to compare polysulfide-treated cowhide glove leather with military glove-shell leather (untreated horsehide) and acrylate-treated horsehide glove leather. Technologists wore gloves or mittens fabricated from the leather for handling wet bricks, lifting, turning, and stacking them from a resting position in approximately 1 inch of water. Figure 2 shows the time required for initial water penetration as well as the rates of water absorption during the test.

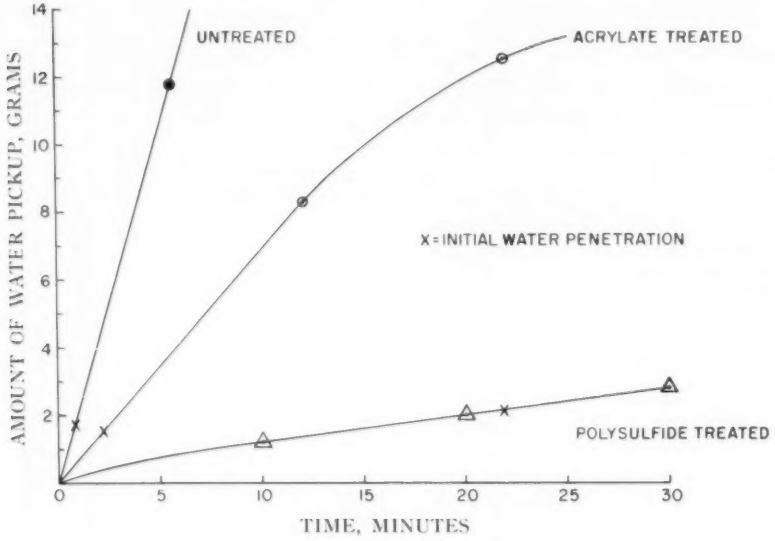


FIGURE 2.—Wet brick test for water resistance of gloves.

The initial water penetration or feeling of wetness was noticed by the technologist in approximately 30 seconds for untreated leather, 1.5 minutes for acrylate-treated leather and 22 minutes for polysulfide-treated leather. Differences in water resistance are also shown by the rates of absorption. In less than 10 minutes palm areas of the untreated or acrylate-treated leather were saturated with water. In contrast, the polysulfide-treated leather retained its dry feel after 30 minutes, except in small areas of the palm where the leather had been squeezed tightly against the wet bricks. It also was comparable or superior to untreated leather in other properties, such as drying time and flexibility after soaking.

Since no standard method was available for measuring resistance of leather to POL products, it was necessary to devise a suitable laboratory procedure. Guided by experience in water-resistance testing, initial trials were made using the Maeser Dynamic Flex Test, in which the leather specimen was mounted grain side up, forming a trough to contain kerosene. This method proved to be unsatisfactory for glove leather as it did not apply sufficient strain to the leather fibers to force kerosene through treated leather or leather coated with a repellent surface film. For example, specimens that showed resistance to penetration for several thousand cycles in this test sometimes were penetrated by kerosene in less than 1 minute of rubbing with a finger under moderate pressure. A test procedure was devised for measuring the time required for penetration of gasoline through a leather specimen rubbed with a rubber-covered finger. The specimen is placed grain side up on a paper towel resting on a flat surface. Approximately 1 ml. of gasoline is poured onto the grain surface and rubbed vigorously with the rough surface of the rubber finger. The specimen is lifted after 30 seconds, 1 minute, and 2 minutes to see if penetration and staining of the absorbent paper has occurred. The penetration times and corresponding ratings are as follows:

Penetration time (minutes)	Rating
Below 0.5	Poor
0.5 to 1.0	Fair
1.0 to 2.0	Good
Above 2.0	Excellent

When polysulfide-treated pieces containing 9 to 24 grams polymer per square foot of leather, referred to in Fig. 1, were tested for gasoline resistance, they rated excellent over their entire area after impregnation at the 15-gram level or higher. Excellent ratings also were obtained on the 10 tannery-treated sides referred to in Table III.

The POL resistance of polysulfide-treated cowhide glove leather also was evaluated in a field-wear test comparing treated and untreated leather mittens. The mittens were worn over wool inserts on a specially constructed POL material handling course, in which test subjects performed operations such as filling a 5-gallon can from a large oil drum and wiping off spillage with rags saturated with gasoline. Tests were conducted over a period of 22 days at temperatures as low as minus 31°F. Both mittens and inserts were weighed at intervals during the test to determine the petroleum pickup shown in Table IV.

As Table IV shows, the average pickup was 101 grams for untreated mittens compared to 54 grams for the treated mates that were worn for a much longer period. Many of the untreated mittens developed failures during the wear

TABLE IV

FIELD WEAR TRIAL OF TREATED VS. UNTREATED MITTENS
PETROLEUM PICKUP DURING HANDLING OPERATIONS

(*Average values*)

Mitten	Number of Mittens	Number of Traversals	Original Weight of Mitten Insert, grams	Petroleum Pickup after Test Completion (Mitten and Insert), grams	Petroleum Pickup per Traversal, grams
Treated	24	74	174	54	0.73
Untreated	24	52	168	101	1.94

period and were replaced with nontest mittens to complete the test of the treated mates. The untreated mittens absorbed petroleum products at a rate nearly three times that of the treated ones. At the conclusion of the test the subjects commented favorably on the relatively dry condition of the treated mittens as compared to the poor feel and appearance of the untreated ones. Only the treated mittens were considered satisfactory for resisting petroleum products, although both types were considered adequate in gripping characteristics and hand dexterity.

The maximum amount of polysulfide polymer that may be used satisfactorily without adversely affecting glove leather is determined largely by the effect of the treatment on flexibility. A measure of this effect is the force required to bend a specimen through an angle of 20 degrees, using the Tinius-Olsen Stiffness Tester. The common single-layer specimen of glove leather proved to be too flexible, but a 3″ x 4″ specimen folded and fastened in a four-layer thickness provided a satisfactory measurement. A comparison of polysulfide-treated glove leather with the standard heavy-duty glove leather and with similar commercial extra-heavy-duty glove leather, one ounce thicker than that required in military specifications, is presented in Fig. 3.

Compared to the regular heavy-duty leather, the polysulfide-treated leather containing a polymer concentration of 18 grams per square foot of leather proved to be more flexible at room temperature and at minus 20°F., perhaps because of a lowered concentration and more uniform distribution of the oils within the treated leather. At minus 40°F. the difference in stiffness is probably not significant. It may be observed that the thicker commercial leather is much stiffer than the other two under all conditions, the difference being very significant at the two lower temperatures.

The effects of polysulfide concentration on stiffness of cowhide glove leather at different temperatures are shown in Fig. 4. Each point on the curves represents the average value obtained on five specimens.

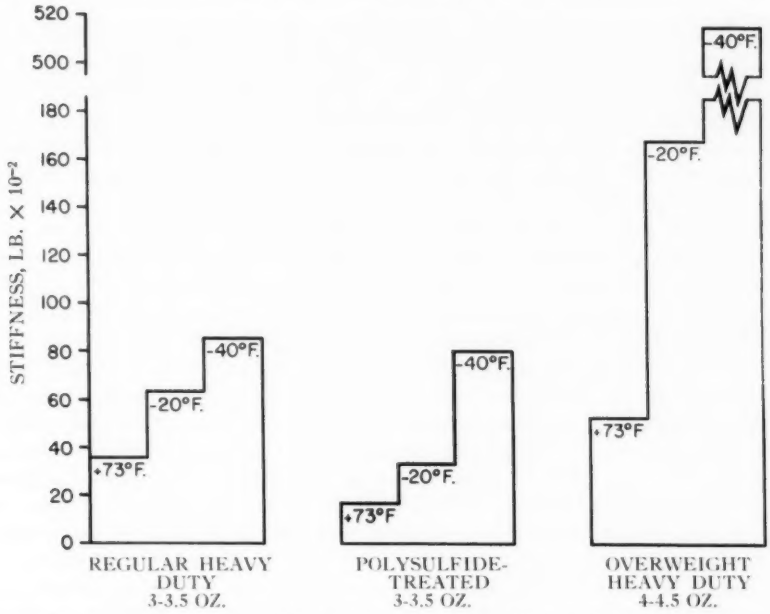


FIGURE 3.—Stiffness for treated and untreated cowhide glove leather vs. temperature.

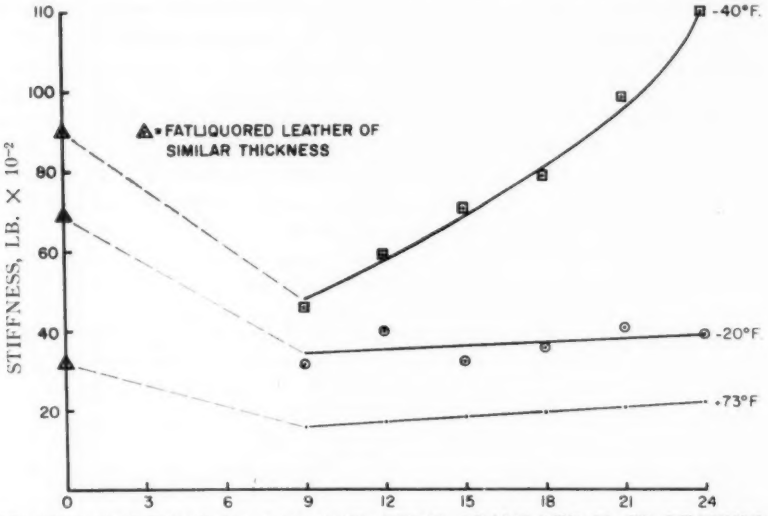


FIGURE 4.—Stiffness for cowhide glove leather at different polymer concentrations vs. temperature.

The results show that the stiffness of the polysulfide-treated leather does not exceed that of untreated leather until the treatment level reaches 21 grams or more. Moreover, low temperatures cause no significant increases in stiffness until minus 40°F. has been reached.

It is generally impossible to determine precisely whether a leather treatment will affect feel or comfort of a glove, but physical properties such as absorption and permeability to water vapor are definitely related to comfort. Leather treated with polysulfide at the 18-gram level absorbs water on the flesh side almost as readily as untreated leather and its permeability to water vapor is approximately 300 g. per square meter in 24 hours. This value is considered to be satisfactory for comfort. Limited wear trials, including the POL-handling test described above, appear to confirm this conclusion.

Water-resistance properties of leather may be adversely affected by leaching or migration of the treating material in storage or use, especially under severe conditions such as repeated wetting and drying. Because of the reaction converting the polysulfide polymer to a more insoluble material, it would be expected to resist migration or washing once the material is deposited in the structure. A test was made to determine the effect of repeated washing on polysulfide-treated leather. Specimens from five sides of tannery-treated cowhide glove leather were laundered 10 times in succession. Each time the leather was washed with laundry soap for 15 minutes at 140°F., rinsed, and then dried at 140°F. for 30 minutes. When the leather was tested afterward, all specimens resisted water penetration for more than 600 cycles and maintained their excellent rating for gasoline penetration.

The polysulfide-treated leather has shown promising results in resisting many different liquid chemicals. For example, the treated cowhide glove leather has been tested for resistance to battery acid (34% sulfuric acid), ethylene glycol, and 2-normal sodium hydroxide solution. In each case 10 grams of liquid were poured onto the grain surface and allowed to stand from 15 to 60 minutes. Comparison of bursting strength measurements for the exposed and adjacent areas showed no loss in strength except exposure for one hour to the sodium hydroxide solution.

DISCUSSION

Application to glove leathers.—The formulations of the treating solution described in Table II and the method of application used in most of the experiments discussed above were satisfactory during laboratory studies and presented no difficulty in the tannery trials. However, changes in the formulations may become necessary in some instances; for example, some leathers contain resinous or synthetic tanning agents that, as mentioned before, tend to act as anticatalysts and require a higher than usual concentration of morpholine for an effective cure.

It was found convenient in this study to follow a standard procedure of degreasing leather prior to treatment in order to eliminate the variable effect of excess oil. Degreasing is not an essential step in the process, however, as the polysulfide treatment has been applied successfully to leather containing more than 10% oil. For regular production of polysulfide-treated leather, a lower oil content is suggested. There are indications that a lower oil concentration provides adequate lubrication with generally more satisfactory results. Leather properties such as density, insulation, feel, and flexibility are adversely affected, especially in cold weather, by large amounts of oils; therefore, any excess should be avoided. A remarkable feature of the polysulfide polymer treatment is its firm fixation within the leather which allows the leather to be processed by regular tannery washing and fatliquoring after the polysulfide treatment without affecting resistance to gasoline or water penetration.

The polysulfide treating materials described in Table II do not present a serious hygienic problem in use, but should be handled in a well-ventilated area using normal precautions. Protective aprons and gloves are recommended to avoid prolonged contact with the skin. The flash point of the treating solutions (approximately 170°F.) indicates that they are not a serious fire hazard.

One objectionable feature found in certain lots of polysulfide-treated leather was an undesirable odor which persisted for several weeks or months despite repeated attempts to eliminate it by heating and washing. In the earlier experiments, most of which were performed on leather from only one tannery, the problem of odor appeared to be a limiting factor in the acceptability of polysulfide-treated leather. Much more favorable results were obtained, however, when the treatment was applied to leather from several other tanneries. These lots did not develop an odor, or in a few cases developed only a slight but acceptable odor. There are indications that the variation in results may be dependent upon the materials used in the fatliquor. In any case, the elimination of the odor appears to be simply a matter of minor changes in materials applied during the tanning or fatliquoring operation.

The polysulfide polymer treatment appears to offer several advantages over other similar processes, particularly with regard to the combination of properties it imparts to glove leather. Since polysulfide polymer-treated cowhide glove leather is capable of resisting water, POL products, and some liquid chemicals, like battery acid, such leather eventually is expected to allow a single multipurpose military glove to be issued.

Experimental applications of the polysulfide polymer to other soft types of leather, such as horsehide, sheepskin, and deerskin, indicate that treatments similar to those applied to cowhide produce good results. Water resistance as high as 600 cycles in the tapping test has been attained as well as excellent resistance to gasoline. However, the relatively loose fiber weave

of these types of leather presents a more difficult problem than the more compact structure of cowhide leather; accordingly, they may require a slight change in formulation or a higher treatment level for effective results.

Although most of these applications were made on chrome-tanned leather, the polysulfide treatment has also been applied successfully in a few tests to glove leathers of other tannages, including vegetable and various experimental tannages.

Studies of storage stability are under way on glove leather specimens treated with various solutions, including some in which plasticizers and chain-stopping agents were incorporated. These studies are most important for military items which may be stored for two years or more prior to use. Gloves and leather specimens which were treated over three years ago with some of our initial treating solutions have shown no apparent change in feel or flexibility.

Application to shoe upper leather.—After completion of the major portion of the laboratory work on producing a liquid-resistant cowhide glove leather, efforts were directed toward producing shoe upper leather of similar characteristics. The formulations developed for glove leather, for instance, solutions A and B of Table II, were immediately found less suitable for upper leather. In one preliminary trial, though, the effectiveness of the liquid polysulfide polymer Thiokol LP-32 was demonstrated by carrying out a wading test with a boot that had a rubber sole directly molded to polymer-treated upper leather and the seams all tightly sealed. After seven hours of walking with the lower portion of the boot in water, a technologist found the inside of the boot and sock "bone-dry". Despite this encouraging beginning, further work showed that additional laboratory studies and tannery trials are yet required.

A recent tannery trial was made on 10 sides of corrected-grain, chrome cowhide Army upper leather with what appeared in laboratory tests to be a promising formulation, consisting of 100 parts Thiokol LP-32, 50 parts THFA, and 10 parts morpholine. The dyed but unfinished leather received 30 grams of polymer per square foot, which is considerably more than what was required for glove leather. Application was the same as for Solution A of Table II. The sides of leather were finished by the usual tannery steps and materials without difficulty.

The leather produced in this trial is normal in appearance and feel. It does not have a peculiar odor or any other undesirable features. Its POL resistance is very good, but its water resistance is no more than fair.

Results like these are encouraging, but at the same time they prove that further work is needed. Different tannages produce leather varying greatly in absorptive properties and, therefore, requiring a variety of techniques. Double treatments, for instance, have been tried out on a small scale with considerable success.

ACKNOWLEDGMENT

The authors wish to thank the personnel of the A. C. Lawrence Leather Company, Peabody, Massachusetts, where several pilot-tannery trials were conducted, and especially M. Battles and J. Bassett, for their cooperation. The assistance of Pfc. E. Welch in the performance of the physical tests is also acknowledged.

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Received August 25, 1958.

DISCUSSION

DR. SHU-TUNG TU (United Shoe Machinery Corporation): There are several kinds of polysulfide polymers. My first question is: Could you tell me which one you think is suitable or that you have found satisfactory for glove leather?

The second question is: What is the thickness of the film and what is the penetration of the polymer?

Third, what is the history of the application of the polymer? (This is glove leather.) At what stage do you apply it? Is it chrome-tanned leather? How about the dye application?

MR. MANN: We applied Thiokol LP-32 and found it more satisfactory than other polymers available at the time we started this work. We have not tested those other products exhaustively.

As far as penetration goes, we think we get penetration approximately a third of the way through from the grain side. We know the flesh side absorbs water or perspiration readily, and when we recently made bending tests of the leather at extremely low temperatures, we noticed the position of the break which indicated that the Thiokol penetrated about a third of the thickness.

The treatment is to be applied after the dye. I think you would run into difficulty dyeing this kind of leather. It can be applied to some types of finished leather, although in general that would not be the way the tanner

would apply it. It could be applied after fatliquoring, or as we have shown here, you can fatliquor afterward, if it is desirable.

DR. TURLEY (Rohm & Haas Company): Is everything O.K. from the toxicity angle and the question of skin dermatitis and so on? Would it be safe to use?

MR. MANN: The morpholine catalyst in this case is the kind of chemical that you have to be careful with. We would recommend that you take normal precautions in treating. However, I do not think it is extremely dangerous in the low concentration used here. We have not determined toxicity, however.

MR. CASSEL (National Bureau of Standards): I did not understand why odor was not more of a problem. You would expect it to be, but you apparently did not have any problems along this line. Is it because of the uniqueness of this particular Thiokol product?

MR. MANN: No, we think it has to do with the oils that are used in the leather. The first leather we used was a garment type which did cause some difficulty in eliminating odor. However, we had no difficulty with upper leather and no difficulty with certain tannages of glove leather.

DR. MELLON (Eastern Regional Research Laboratory, USDA): Did you attribute the differences you found from side to side to the mechanical treatment of application or to differences in the leather?

MR. MANN: We did not investigate that exhaustively. It probably was due partly to variation in hides. Some hides may be a little more absorptive and require a little more material. However, these men were inexperienced, and it could be partly due to uneven application.

DR. WALLACE WINDUS (Eastern Regional Research Laboratory): These materials are domestically produced, I realize, but the same types of compounds are solid propellants for rockets. I wonder about the availability of them to the leather industry, in an emergency.

MR. MANN: There has been no indication of any shortage whatever. I don't think the same materials are used.

JOSEPH ORIETAS (Albert Trostel & Sons Company): How would such a treatment affect the subsequent finishing of the leather?

MR. MANN: Undoubtedly there will be a problem in applying the normal-type finishes, as with any other water-repellent treatment. The only experience we have had is with one special type of finish, applied not in the normal way but from solvent-type solution. There was no difficulty whatever with that one. That I would not consider a typical finish.

LIFE LINES

Brief Biographies of Our Contributors

JOSEPH C. BARRY received a B.S. in Chemistry from Providence College, Providence, R. I., in 1950 and his M.S. in Physical Chemistry from the Catholic University of America, Washington, D. C., in 1955. Following a period of employment as operator of a dairy company control laboratory, he joined the staff of the Leather Technology Laboratory Branch of the U. S. Army Quartermaster Research and Engineering Command, Natick, Massachusetts, in 1955 and became an ALCA member in 1958.

ROBERT B. COMBS, JR., received his B.S. degree from the University of Kentucky, Lexington, Kentucky, in 1951. From June 1, 1951, to June 1, 1954, he served as a chemist for the Chrysler Corporation, Detroit, Michigan, in the analytical chemical control laboratory. He joined the staff at the Tanners' Council Laboratory on November 1, 1954, as a research associate and served in this capacity until March, 1958. He worked on special projects concerned with the chemical modification of leathers, on chemical and physical testing of calfskins and leathers, and on modification of the elastic tissue of animal skin.

EUGENE C. DOGLIOTTI received a Bachelor's degree in Chemical Engineering from Drexel Institute in Philadelphia, Pa., in 1944. He did graduate studies at the University of Pennsylvania from 1947 to 1949. He joined the Leather Technology Laboratory Branch of the Quartermaster Research and Engineering Command in 1950 and became a member of ALCA in the same year.

CHARLES W. MANN, whose biography was published in May, 1956, is now Chief, Leather, Footware and Handware Branch, Textile, Clothing and Footware Division, Q. M. Research and Engineering Command.

DR. JOSEPH NAGHSKI—See *JALCA*, February, 1958.

WILLIAM T. RODDY—See *JALCA*, June, 1958.

To Improve Hides and Skins. The depilated and cleansed skins are placed in a fluid compounded with glycerine, and allowed to remain until thoroughly saturated. This will require from 1 to 4 days, according to the thickness of the skins. They are then taken out, freed from the excess of glycerine, dried, and stored away for future use. Skins moderately tanned can also be subjected to the same operation. Skins thus prepared can be advantageously used for machine belts, straps, etc.
—*The Techno-Chemical Receipt Book.* Philadelphia: Henry Carey Baird & Co., 1903.

ALCA NEWS

Proceedings, Reports, Notices, and News

of the

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JOURNALS FOR SALE

The following journals are being offered for sale by the Orthmann Laboratories, Milwaukee, Wisconsin:

Industrial and Engineering Chemistry. Vol. 43–48 (bound) and 49–50 (not bound).

Analytical Chemistry. Vol. 23–28 (bound) and 29–30 (not bound).

Chemical Abstracts. Vol. 45–49 (bound).

JALCA. Vol. 1–48 (bound).

JOINT ALCA-ASTM COMMITTEE ON LEATHER

SECTION IV—MEASUREMENT OF WATER RESISTANCE

Meeting November 13, 1958, Philadelphia, Pa.

A method for Water Absorption, Static, was submitted to Dr. Lollar for action by his committee but was retracted later in the day, pending action by our group on a suggested change in the method of calculation from a weight to volume basis in order to present a clearer picture of the water absorption of light-gravity leathers.

The alternate methods for Water Penetration, Dynamic, were discussed but are still held up pending further evidence concerning work done by Messrs. Lollar and Maeser.

Dr. Chouinard reported that his subcommittee had found the Spray Test to give reasonably concordant results as regards surface characteristics of garment leather. This test would be written for use on leather substantially as it is now written for textiles. Their method for water absorption on whole skins looks promising and is now undergoing correlation tests in different laboratories.

C. W. Mann reported that the Tap Tester has given satisfactory results on three of five commercial lots of glove leather, and samples will soon be submitted to other laboratories for correlation.

Dr. Kanagy reported the results on Dynamic Water Penetration on samples of crust, finished, and impregnated sole leathers from two sources. Arthur Kay is collecting further samples of leathers which will be tested first by Dr. Kanagy and later by Kay and Wagoner on the same apparatus.

Messrs. Kanagy, Mann, and Cooke have volunteered to explore further the Kubelka Method for Static Water Absorption as soon as we locate a source of the apparatus used.

Bill Cooke volunteered to furnish some preliminary data regarding the dimensional stability of leather after wetting and drying.

The committee recognized the need of a method for measuring the water penetration of finished shoes, particularly those with vulcanized bottoms. Considerable discussion brought out the fact that the Dow Corning Tester has not correlated well with wear tests, and the degree of flexing will be further investigated by Messrs. Dudley and Mann.

R. G. Ashcraft
Chairman

CANDIDATES FOR THE 1959 ELECTION

CANDIDATES FOR COUNCIL

James E. Byron
Frank Edmonds
C. Paul McKee
Elwood E. Presley
Andrew Salamatov
Herbert A. Tetreault

The above slate of candidates has been prepared by the Nominating Committee.

Anthony J. Pilar, Jr.
Robert L. Young, Jr.
Adam M. Lammert, *Chairman*

Members note:

Additional candidates may be nominated by petitions signed by 15 or more active members. Such petitions shall reach the Secretary not later than April 1, and shall contain a statement of the willingness of the nominee to serve if elected. No member shall sign more than one petition for each office to be filled.

NIELS BJERRUM

1879–1958

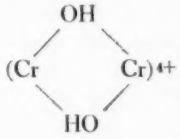
The death of Professor Niels Bjerrum, the renowned Danish chemist, which occurred on September 30, 1958, in Copenhagen, means the loss of one of the outstanding personages of chemistry, particularly physical chemistry and more specifically solution theory and the chemistry of complex inorganic compounds.

Since two major fields of research in Bjerrum's wide scientific inquiry have had and still have, most profoundly so, a direct bearing upon leather chemistry, Niels Bjerrum's name belongs to the science of leather chemistry also. His outstanding research on the fundamentals of the chemistry of basic chromium salts and the physicochemical behavior of ampholytes, to which proteins and thus also collagen belong, has contributed substantially to our present conception of the chrome tanning process. Actually they form the basis for the modern concept of this involved reaction. An expression of our intellectual debt to the departed scientist seems therefore fitting.

Niels Bjerrum, who came from a well-known Danish family of scholars, was born in 1879. His first major contribution to chemistry was his inaugural Dissertation submitted to the University of Copenhagen in 1908. This comprehensive work, with the original title: "Studier over basiske Kromforbin-

delser", *i.e.*, "Studies on Basic Chromic Compounds", is now a classic in its field. The nature of the protolysis of chromium salts, mainly chlorides and sulfates, was for the first time established on the basis of modern physical chemical theory. Experimental evidence for the formation of polynuclear chromium complexes by the protolysis of solutions of chromium salts was supplied by determination of reaction constants. Bjerrum proved that on protolysis of chromic chlorides the unstable hydroxo-compounds were first formed and subsequently transformed into the stable dinuclear chromium compounds now known under the name of Ol-compounds.

By addition of alkali to solutions of chromium salts the primary formation of the simple hydroxo-compound, $(Cr-OH)^{2+}$, was also shown to be an intermediate step to the final protolytic product, the diol-compound



A few years later Pfeiffer was able to prepare derivatives of such polynuclear chromium compounds, named by him Ol-compounds. During the 1920's Stiasny further extended this line of research to Ol-compounds of importance in tanning. Bjerrum's *magnum opus* in chromium chemistry was published in Danish. Although a detailed account was given in German translations (*Z. phys. Chem.*, 1910), his fundamental contributions first received their due recognition some twenty years after their appearance.

Bjerrum's interest came later to be directed mainly to solution theory, particularly to the physical chemistry of strong electrolytes. In this field Bjerrum's name is of great renown. He was the first to present conclusive evidence for the concept of the complete ionization of strong electrolytes, advanced the concept of ion pairs and prepared the ground for the coming of the Debye-Hückel theory.

However, the complex chromium salts which had been the object of his first independent investigation, were not to be forgotten. On several occasions he returned to the love of his youth. Most noteworthy is his model study of the thiocyanates of chromium, in which the complex constants of the various molecular species were determined (1913). The experimental proof of the presence of all the six possible complexes in the solution of the hexa-thiocyanate and the estimation of the complexity constants of these compounds are outstanding feats, still unmatched in complex chemistry. It should be mentioned that the study of the protolysis of the chromium salts gave the impetus to the creation of the present concept of protolysis ("hydrolysis") and to the formulation of the acid-base concept, now generally accepted, which usually

is associated with the name of J. N. Brönsted, Bjerrum's schoolmate and later "professorial" competitor.

Bjerrum's studies of ion pair association and dipoles were instrumental in leading to his subsequent enquiry into the nature of ampholytes. The results are laid down in his classical paper of 1923 on the zwitterionic behavior of amino acids. The pK values of the amino acids that were then accepted—the acid as well as the basic ones—did not conform with the structural formulation of the amino acids prevailing at the time. Bjerrum supplied an array of experimental evidence for the present concept of the nature and reactivity of amino acids and proteins, the ampho-ionic theory. The presence of dipolar molecules of the general form: $COO \cdot R \cdot NH_2^+$ in water solution in the pH range corresponding to the isoelectric points of the ampholytes was established. This concept forms the basis of our present view of the mechanism of the principal reaction in the chrome tanning process.

In 1925, in a paper presented by the present writer before the American Chemical Society at the Baltimore meeting, the Bjerrum "zwitter ion" idea was first applied to reactions of proteins. It was stressed that the fixation of cationic chromium complexes by collagen from solutions with pH values on the acid side of its isoelectric point was logically explained by the zwitterion theory of Bjerrum, the positively charged chromium complexes competing with the protons for the possession of the charged carboxyl groups of collagen (*Ind. Eng. Chem.*, **17,** 577 [1925]).

Professor Bjerrum was long unaware of the fact that he had supplied the leather chemist with the fundamentals on which our present conception of the mechanism of chrome tanning rests: (1) the polynuclear nature of basic chromium salts, as to the tanning agent, and (2) collagen reacting as a typical zwitterion, as to the substrate. It became my pleasure to inform him about this pleasant fact on a visit with him some thirty years ago. On later occasions he was present at meetings of the Nordic leather chemists in Copenhagen, being honored as the grand old man of chromium chemistry.

It must have been a source of great joy for him to experience the superb scientific acumen of his son, Jannik Bjerrum, professor of inorganic chemistry in Copenhagen, who is perpetuating the Bjerrum tradition in the physical chemistry of complex compounds.

K. H. Gustavson

To Preserve the Yelks of Eggs used in Tawing Glove Leather. Rub fine in a mortar or upon a stone 1 pound of yelks of eggs, ¼ ounce of common salt, and ¾ ounce of starch. The mixture, on thickening, is poured into moulds and dried in the air. Yelks of eggs thus prepared answer the same purpose as fresh.
—*The Techno-Chemical Receipt Book.* Philadelphia: Henry Carey Baird & Co., 1903.

ABSTRACTS

Second International Colloquium of Leather Technologists. Miroslav Tomisek. *Kozarstvi*, **7**, 4–6 (1957).—This conference at Otrokovice, Czechoslovakia, Oct. 8–13, 1956, attended by technologists from eastern Europe, China, and Mongolia, was devoted to the production and use of syntans. The following conclusions were stated in a resolution passed: All kinds of leather of good quality can be made with exchange syntans as a component of the tanning blend. A high percentage of auxiliary syntans tends to lower the quality of the leather. Lignosulfonate cannot be considered a tanning material and should not be considered as a component of the tanning blend. Production of exchange syntans from lignosulfonate does not seem to be economically possible at present. The best raw materials for exchange syntans are monohydroxy phenols, naphthols, and especially pyrocatechol and resorcinol. The tanning properties of syntans vary inversely with the $-SO_3H$ content, not only of the syntan itself but also of the blend as a whole. For this reason, when a large amount of exchange syntans is used, the use of sulfited vegetable extracts should be restricted, and the total tannin that is given should be increased. The hide powder method does not give the correct value for the tannin content of exchange syntans, and research to find a better method is needed. Adherence to the International Union of Leather Chemists' Societies was recommended. The next colloquium, on the theme "Finishing of Leather," will be held either in Budapest or Moscow.
L.M.

Roumanian Problems of Obtaining a Better Quality of Indigenous Tanning Materials and Reducing Their Importation by Use of Syntans. G. Havas and N. Sucitulescu. *Kozarstvi*, **7**, 163–65 (1957).—The principal Roumanian tanning materials are spruce and oak barks and oak wood. If importation of tannins should be forbidden, spruce would constitute 70% of the available tannin, and a too long tannage would be necessary. Some preliminary attempts were made to condense phenol or cresol with spruce tannin by means of HCHO, but production of syntans proved to be the best solution. The development work is described. Romatan BCF is a condensate of phenol and 2-naphtholsulfonic acid; BD is a dihydroxydiphenylsulfone condensate dissolved in 2-naphtholsulfonic acid; CL is a condensate of ligninsulfonic acid with cresolsulfonic acid and phenol; RL is a phenol-HCHO resin dispersed in a partly desulfonated and demethylated ligninsulfonic acid. Tanning with these Romatans is described and their analyses are given. L.M.

The Application of Romatan Syntans for the Tanning of Sole Leather in Roumania. N. Sucitulescu and I. Dimitrescu. *Kozarstvi*, **7**, 49, 74–75 (1957); *see* preceding abstr.—Production of Romatan syntans, recently started, should amount to 2000 tons of 100% tannin in 1958. Pilot-plant tests with Romatans, in comparison with regular full-scale production in Flacara Rosie tannery, are described. The starting composition of the blend was oak wood extract 45%, chestnut 10%, sulfited quebracho 20%, valonea 10%, spruce bark 10%, and USSR syntan PL 5%. Tannage was done in 8 suspenders, 7° to 38°Bk., 18° to 22°C., pH 5.6 to 4.5, 600% liquor. Layers had 43° to 60°

Bk., 30°C., pH 4.4 to 4.2, 200% liquor. Drums had 70° to 95°Bk., 35° to 38°C., pH 5.0 to 4.2, 250% to 180% liquor. In the pilot-plant tests, from 10% to 40% of Romatan was added to this blend. The permissible limit was found to be 30%, or perhaps 40%, for Cr-vegetable leather. The syntan was added only to the suspenders and layers: the drum liquors contained only vegetable tannin, and therefore a less hydrophilic leather was obtained. L.M.

Dihydroxybenzenes as Raw Material for the Production of Syntans in Czechoslovakia. A. Kala. *Veda a Vyzkum v Prumyslu Kozedelnem*, **3**, 53–61 (1958).—Analyses of the dihydroxybenzene fraction of the middle oil fraction of soft coal tars show that this material has a very variable composition. "Difen", a commercial product, gave 5% distillate to 248°C. and 95% to 268°C. It contained pyrocatechol 26%, 4-homopyrocatechol 22%, 3-isopyrocatechol 8%, and other higher phenols 15.5%. Omega syntan is made from 3 mols Difen, 3 mols HCHO, and 1 mol Na_2SO_3. Syntan OM_2 is made from 3 mols Difen, 2 mols HCHO, H_2SO_4 as catalyst, and 1 mol Na_2SO_3. Omega syntan contains tannin 41.6%, nontannin 4.6%, and has combining values of 44.5 and 48.1 at 3 hr. and 24 hr. respectively. OM_2 has combining values of 50.5 and 53.6. A new syntan, P2, has been made on a pilot-plant scale from technical pyrocatechol and HCHO; it contains no SO_3H groups. It contains 69% tannin and only 0.8% nontannin, and gave combining values of 70.9 and 78.8 at 3 and 24 hr. In contrast, K_2D, a widely used syntan, contains 35% tannin, 6% nontannin, and gave combining values of 53.3 and 62.1. Leathers tanned with mixtures containing 50% of either P2 or K_2D had the following characteristics:

Syntan	Degree of Tannage	Percent Water Absorbed in 2 hr.	Percent Water Absorbed in 24 hr.
P2	88.7	28.7	35.7
K_2D	70.3	34.8	43.3

The differences are ascribed to the SO_3H groups in K_2D. L.M.

Present State of Production and Research of Exchange Syntans in Czechoslovakia. I. Binko. *Veda a Vyzcum v Prumyslu Kozedelnem*, **3**, 5–23 (1958).—The production of exchange syntans in Czechoslovakia was started because of difficulties in importation of vegetable tannins and declining production of domestic spruce bark (*see JALCA*, **53**, 667 [1958]). The percentage of syntan used in the tanning blend had to be reduced from 32% in 1953–54 to 25% in 1955–56 because of lower quality of sole leather. Spruce constituted 28.5%, 30%, 30%, and 33% of the tannin used for sole leather in 1953, 1954, 1955, and 1956 respectively and imported tannins constituted 39.5%, 38%, 45%, and 42%. In 1956, tanners used 10% of syntans K_2D/K and SP/K, 3% of K_1D/K, and 12% Kortan Q_1 on the pelt weight. Raw materials for syntans are discussed. Syntans made from raw, turbid phenols from soft coal tars have a "K" after their symbols. The large production of naphthalene in Czechoslovakia permits the production of 2-naphthol. The Stalin Works at Zaluzi produces very large quantities of dihydroxybenzenes, largely pyrocatechol

and its homologs, sold as "Difen" or as a purer fraction called "Optol" (*see* preceding abstract). Ligninsulfonates are a very poor base for syntans; therefore, production of a syntan of the Tanigan extra A type has not been introduced, even if such production has been satisfactorily accomplished in the pilot plant. The consumption of K_1D/K, used only for filling sole leather, is decreasing. The quality of foreign syntans is discussed. The tendency in Czechoslovakia is to produce only the best syntans, eliminating the older brands, to eliminate syntans based on pure phenols and ligninsulfonates, and to enlarge the consumption of Difen and Optol. Consumption of materials in percent "effective weight" of the various syntans is as follows:

	SN_{25}	K_2D/K	K_1D/K	SP/K	BNO	Kortan Q_1 (solid)	Kortan QD (solid)
Phenol	32.7	—	—	—	—	—	—
Crude phenols from soft coal tar	—	20.1	5.0	160	—	—	—
"Difen"	—	10.1	6.25	—	—	34.8	—
"Optol"	—	—	—	—	—	—	36.5
Phenol-cresol mixture	—	—	7.5	—	—	—	—
Ligninsulfonate (50% solution)	—	—	28.1	—	—	—	—
o-cresol	—	—	—	—	17.0	—	—
2-naphthol	—	—	—	—	13.0	23.8	20.7
HCHO (40%)	26.8	19.8	13.4	20.7	13.5	25.7	26.45
H_2SO_4 (92.5%)	10.1	8.3	4.75	9.2	—	—	—
H_2SO_4 (100%)	—	—	—	—	12.5	22.6	20.0
Acetic acid (100%)	—	—	—	—	—	2.56	1.46
$Al_2(SO_4)_3 \cdot 18H_2O$	—	—	—	—	—	3.93	—
NaOH	3.8	3.7	2.35	3.5	—	—	—
Ammonia (25%)	—	—	—	—	11.5	19.5	9.1
Tons produced (100% tannin) (1955)	548	668	167	69	20	899	—

Principles of production of syntans are discussed. Sulfonic acid groups should be kept to a minimum; perhaps a practical limit has been reached in SN_{25}. The decrease of acidity during tannage of tan liquors containing syntans can be somewhat lowered by neutralizing the syntans with ammonia and then acidifying with acetic acid, but then there is an undue swelling of hides toward the end of the tannage. The next goal of research is to produce syntans without any $-SO_3H$ groups. These groups are a nuisance also in sulfited spruce and even in sulfited quebracho extract. It is not advisable to duplicate German products but rather to follow an independent line of research. Another goal of research is to produce syntans that are less expensive than vegetable tannins. Only the necessity of disposing of crude phenols from soft coal, together with the availability of Difen, makes it possible to market K_2D/K and SP/K at lower prices than vegetable tannins. L.M.

Solvent Tannage [with Syntans]. H. Weber, *Kozarstvi*, **7**, 88–91 (1957).—A new syntan, made from 6 mols dihydroxybenzenes and 1 mol

phenol condensed with HCHO and sulfonated, was soluble in water, but the viscosity was too high for it to be used in tanning. Products insoluble in water but soluble in alcohol have been prepared. Their alcohol solutions have good tanning properties. These products, called A-syntans, are made by condensing "Difen" (*see* two preceding abstracts) with HCHO, using H_2SO_4 as catalysts. Products A_1, A_2, A_3, and A_4 had molecular weights of 574, 1173, 1516, and 1359; of these, A_3 was the best. It contained about 15 molecules of pyrocatechol condensed with HCHO, no SO_3H groups, pH 4.5–5.0, and therefore closely resembled a vegetable tannin. Addition of 10% to 15% alcohol produced a solution having a viscosity similar to a vegetable tannin extract. Light skins can be tanned with A_3 in 3 to 5 hr. and heavy hides in 24 to 60 hr. In a laboratory test, 100 parts of delimed and pressed pelt were tanned in a drum with 40% A_3 and 50% alcohol for 50 hr. By adding water to the spent liquor, 11.8% of A_3 was precipitated and recovered, and 39.4% alcohol was recovered from the liquor by distillation, so net consumption was 28.2% A_3 and 10.6% alcohol on the pelt weight. This experimental leather contained 44% hide substance, and 39% combined tannin, shrank at 90°C., and absorbed 42% water in 24 hr. Pretannage with 0.6% Cr_2O_3, or lowering the pH to 3.8 by adding lactic acid, gave no better results. In pilot-plant tests, considerable losses of alcohol occurred because of heating of the load; this was overcome by adding more alcohol to give a longer float. In the final test, 50 kg. (pressed weight) of pelt was tanned with 20% A_3 and 100% alcohol for 12 hr. The temperature did not exceed 30°C. Then 20% more A_3 was added and tannage was continued for 48 hr. Alcohol recovery was 72%. This leather had degree of tannage 84, leather substance 83%, water solubles 0.8%, yield value 222, shrinkage temperature over 100°C. L.M.

Syntans Used by the Polish Leather Industry. E. Bischoff. *Kozarstvi*, 7, 46–49 (1957).—Polish syntans (Rotanins) correspond to the I.G. "Tanigan extra" or "Gerbstoff" types. Rotanins P, M, and W correspond to Tanigan extra A, D, and B. Rotanins BNS and BN correspond to Gerbstoff Que and KN. Production of Rotanins in 1955 amounted to 2152 tons of 100% tannin, including 1589 tons in BNS, and is expected to be doubled by 1960. Only 15 tons of auxiliary syntans will be produced. The percentages of syntans, on total tannin, currently used are 25.4 for sole, 23.0 for fancy, 12.8 for harness, and 10.9 for vegetable heavy shoe upper leather. These percentages are expected to reach 39.2, 33.0, 25.0, and 10.9% in 1960. Harness leather has been tanned successfully in a pilot plant using 70% syntans. The production of sole leather is described. The hides are limed 3–4 days with 9% CaO and 1.2% Na_2S, and delimed with lactic acid to a 25–33% lime streak. There are 10 suspenders, 7° to 42°Bk., and 2 layers (20 days in each) 42°–49° and 49°–56°Bk. with spruce bark. The drum liquors are 77°Bk. for bends, 56° for shoulders, and 49° for bellies. In 1955–56 30.5% tannin on the pelt weight was used, made up of 8% syntans, 4.5% spruce bark, and 18% vegetable tannin extracts. Of these extracts, quebracho comprised 60%, oak wood 20%, wattle 15%, and valonea 5%. The total tanning time was 70 to 95 days. For fancy leather, mostly pig, total tannin consumption was 17%, composed of 8.5% quebracho, 4.35% wattle, 1% spruce bark, and 4% syntans. Production of extracts from sumac and oak wood is described. Analyses and physical test data on leathers are given. L.M.

The Use of Vegetable Oils in Tanneries. V. Pilc. *Veda a Vyzkum v Prumyslu Kozedelnem*, **2**, 59–68 (1957).—Sulfated castor-oil and olive-oil soap have long been used. Analyses of various vegetable oils are given. The following fatliquors are described: (1) a sulfated oil for sole leather from rape seed 70%, sunflower seed 10%, and refined fat from glue factories 20%; (2) rapeseed oil sulfated with 25% H_2SO_4 at 20°C. for suede, calf, and rabbit furs; (3) a 1:1 mixture of soya and sperm oils sulfated with 25% H_2SO_4 at 26°C. for calf; (4) 9 parts of a sulfated mixture of rapeseed 40%, sunflower 40%, and 20% glue factory grease, plus 1 part mineral oil for side and pig upper. A mixture of sulfated and unsulfated sunflower oil applied to waterproof pig leather caused heating and spontaneous combustion. For reasons unknown, this happened only with pig—not with waterproof steer leather. The difficulty can be overcome by adding an antioxidant, e.g., 0.25 to 1.0% of phenyl-β-naphthylamine. Glazed kid, which developed much spue when fatliquored with sulfated neatsfoot oil, showed no spue after several years when fatliquored with 50% sulfated castor, 25% linseed oil, and 25% elain. L.M.

Tanning by Dicyandiamide Resins. Gyorgo Vago. *Kozarstvi*, **7**, 314–18 (1957).—Resin tannages and the production of dicyandiamide and melamine are reviewed. The Retingans were studied. Results of paper chromatography show that Retingan ZN is almost identical with Tamol NNO, an Na salt of a condensate of naphthalenesulfonic acid and HCHO. Retingan R6, corresponding to Chemtan R6, is a condensate of 1 mol dicyandiamide and 3.4 to 4.5 mols HCHO. The technology of filling leather with R6 is described. On treatment with boric acid the pH of R6 falls from 9.5 to 6.0, and complexes are formed resembling those of boric acid with glycerol, mannite, and phenol. Retingan R6 represents the B-stage of condensation, not the C-stage (see Küntzel, abstr. *JALCA*, **50**, 483–84 [1955]). It is not cationic and does not precipitate with Retingan ZN. During long boiling under reflux, the cationic activity (measured by precipitation with Retingan ZN) increases, precipitation by acids ceases, and a gel is formed by adding alkali. The dipole decomposes and amido- and COOH- groups are formed; the pH falls. For Hungary, a C-phase condensate is said to be more useful than Retingan R6, and Retingan ZN is unnecessary as the C-phase condensate is precipitated by any syntan or vegetable tannin. A C-phase condensate (Plastan) has been produced. It contains 4% to 5% ash on the dry basis (less than Retingan R6). Plastan forms gels after 8 days, but addition of 0.1, 0.5, and 2.0 ml. HCHO to 20 ml. Plastan retards gelation to 10, 11 and 30–40 days, while addition of 5.0 ml. HCHO prevents gelation for more than 9 months. The reason is not known. Addition of HCHO to melamine-HCHO condensates gives similar results. Retanning tests were made with Retingan R6 and with Plastan. The split hides were neutralized with 0.2% $NaHCO_3$ and 0.2% NH_4Cl, then treated with 1% of a sulfated oil and 2% Albatan (an *o*-cresol-urea-HCHO condensate) for 20 min. The skins were then treated in a new bath with 80% water and 5% Plastan at 50°C. for 20 min.; then 1% lactic acid was added, the skins were drummed 10 min. and were finally dyed and fatliquored with 3.7% oil. The retannage with Retingan R6 was done according to the manufacturer's directions. The leathers retanned with either Retingan R6 or Plastan were an improvement over straight Cr leather but were inferior to vegetable retan lather. Therefore production of Plastan has not been started. L.M.

Experiments on the Production of Syntans in Bulgaria. B. I. Valchew. *Kozarstvi*, **7**, 165–67 (1957).—Development of syntans in Bulgaria is described. Some syntans are imported from Hungary and the USSR. Laboratory experiments to produce phenolic syntans and their application to tanning are described. L.M.

A Study of an Acid-catalyzed Polycondensation of o-Dihydroxybenzene with Formaldehyde. Z. Kotasek and J. Strachota. *Veda a Vyzcum v Prumyslu Kozedelnem*, **3**, 25–42 (1958).—The polycondensation of phenolic syntans has been little studied. This work should form a theoretical basis for preparing Syntan P2, free from $-SO_3H$ groups, from technical pyrocatechol and HCHO with HCl catalyst. The condensation has been followed by paper chromatography. An apparatus is described for making several condensations at once. Condensation was done at 60°C. to 0.63, 0.70, and 0.77 degrees, with 15.7 g. pyrocatechol; 6.56, 7.7, and 8.8 g. HCHO (39.2%); 0.86 g. HCl (37%); and 31.0 g. water. Free pyrocatechol was determined by ascending chromatography using CH_2Cl_2 and $CHCl_3$ (2:1) as solvent, 3% phosphomolybic acid in aqueous alcohol and ammonia vapor as developer, and estimating free pyrocatechol densitometrically. Condensation practically ceases after 6 hr. Pilot-plant P2 contains about 15% free pyrocatechol, while laboratory preparations condensed with 0.55 and 0.70 mols HCHO contain 40.5% and 28.5%. The condensates have been fractionated chromatographically on a polycaprolactam column, eluted with water-saturated tert.-butanol. The free pyrocatechol is adsorbed preferentially. Column chromatography on basic "MFD Annex" gave the same results and can be used commercially to remove free pyrocatechol from P2. A colorimetric method is described for determining free pyrocatechol with phosphomolybdic acid at pH 3.5, with an accuracy of ± 3%. L.M.

Influence of Syntans on Sludge Formation in Tan Liquors and on Physical Properties of Leather. R. Exner and J. Kabrt. *Veda a Vyzcum v Prumyslu Kozedelnem*, **3**, 63–77 (1958).—Because the syntans used in previous work are obsolete, formation of sludge in tan liquors was restudied using a more refined method. Nemec (abstr. *JALCA*, **32**, 342 [1937]) showed that the sediment becomes irreversibly less colloidal on cooling; therefore, tan liquors were diluted with water at 0°C., which gives higher but more reproducible values for sludge. Statistical analysis gave coefficients of variation for the sludge determination of ± 19.7% for most tannins (± 12.4% for wattle), while the determination of insolubles by the filter method has a coefficient of variation of ± 45%. The components of the tan liquors were sulfited spruce bark, sulfited quebracho, sulfited oak wood, wattle, and chestnut extracts, and syntans used were SN_{25}, K_2D, Omega, Kortan Q_1, and Kortan QD. Stock solutions of tannins and syntans were prepared to contain from 18% to 32% tannin, and 120-ml. portions were diluted to 1 liter with water at 1°–2°C., stored overnight, and filtered. Binary mixtures of vegetable tannins contained up to 50% more sludge than the calculated values. By adding syntans to vegetable tannins, the mean value for sludge falls to 70% for SN_{25}, 40% for K_2D, 75% for Omega, 68% for Kortan Q_1 and 88% for Kortan QD. In the case of sulfited spruce bark, only K_2D lowers sludge to 35% and Kortan Q_1 to 50%; other syntans have no effect. The sludge from ternary tannin mixture is low-

ered only by K_2D. Kortan Q_1, and, in part, Omega syntan. Mechanical properties of leathers tanned with blends containing 32% and 50% syntan in the total tannin are not very different, but the tear strength of leather tanned with a blend containing only 20% syntan is 9% lower. Degree of tannage is lower for 50% than for 32% syntan, and water solubles are at a minimum for 32% tannin. Leather tanned with sulfited spruce bark extract plus 30% syntan had a distinctly lower tear strength and contained 16% more water solubles than leather tanned with unsulfited spruce bark extract and 20% syntan. Leather tanned with sulfited spruce and 50% syntan had 11% lower tear strength, 13% higher degree of tannage, and 11% less water solubles than leather tanned with unsulfited spruce and 50% syntan. These variations are calculated from coefficients of variance, but they would have more validity if based on more analyses.

L.M.

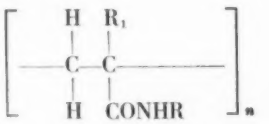
PATENTS

An Alkoxy Aluminum Chelate, a Dispersion of It in an Organic Liquid, and a Water-repellant Porous Object. U. S. Pat. 2,839,421. Charles J. Albisetti, McDaniel Heights, Del., assignor to E. I. du Pont de Nemours & Company, Wilmington, Del., a corporation of Delaware. Appl. April 6, 1955.—2. An alkoxy aluminum chelate composition having the average empirical formula $(R)_xAl(OR')_y$ wherein R′ is alkyl of 1 to 4 carbon atoms, R is the anion of a bidentate chelating agent having one acidic hydrogen and containing an alkyl straight chain of at least 12 carbons, said anion being linked to aluminum through oxygen and at most one nitrogen, x and y are each at least 0.5 and together total 3. Use of these compounds was demonstrated in waterproofing bricks, leather, cotton and paper.

Synthetic Resin Tanning Agents Containing Alkali-pretreated Sulfite Waste Liquor and Process of Preparing Same. U. S. Pat. 2,857,346. Gustav Mauthe, Opladen, and Rudolf Fingado, Leverkusen, Germany, assignors to Farbenfabriken Bayer Aktiengesellschaft, Leverkusen, Germany, a corporation of Germany. Appl. Jan. 21, 1955. Claims priority, appl. Germany Jan. 26, 1954.—1. A process for producing solid kneadable tanning resins which comprises precipitating a mixture of (1) a member selected from the group consisting of a condensation product of (*a*) an aromatic sulfonic acid with (*b*) an aromatic hydroxy compound and (*c*) an aldehyde, and a condensation product of (*a*) an aromatic compound containing at least one sulfo-methyl group with (*b*) an aromatic hydroxy compound and (*c*) an aldehyde, and (2) an alkali-pretreated sulfite waste liquor by means of a member selected from the group consisting of sulfuric acid, hydrochloric acid, an alkali metal salt of said acids and an ammonium salt of said acids.

Process for Precipitating Tannins and Proteinaceous Materials from Liquid Media. U. S. Pat. 2,860,987. Jesse Werner, Holliswood, N. Y., assignor to General Aniline & Film Corporation, New York, N. Y., a corporation of Delaware. Appl. Oct. 31, 1957.—1. The process of precipitating substances selected from the group consisting of natural tannins and protein-tannin com-

plexes from liquid mediums containing the same which comprises adding to said liquid medium, in a precipitating amount, a polyacrylamide having the following general formula:



wherein R represents a member selected from the group consisting of hydrogen, methyl and ethyl goups, R_1 represents a member selected from the class consisting of hydrogen and methyl and *n* represents the extent of polymerization indicative of a molecular weight ranging from 100,000 to 2,500,000.

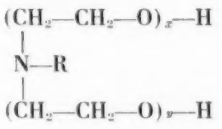
Process of Dyeing Wool with Vat Dyes. U. S. Pat. 2,857,235. Salvatore G. Coscia, Philadelphia, Pa., assignor to the United States of America as represented by the Secretary of the Army. Appl. July 19, 1954.—1. A process of dyeing wool fibers, comprising contacting said wool fibers for a period of from about 10 to about 60 seconds with a bath containing dissolved therein an anthraquinone vat dye, alkali hydrosulfite and caustic alkali, the amount of said caustic alkali being regulated to maintain an electrometrically determined pH value of not less than about 12.8 and not more than about 13.1 in said bath, the temperature of said bath being between about 120° F. and about 160° F., so as to effect deep-dyeing of said wool at its point of maximum affinity for said vat dye and before fiber damage occurs at said pH value, and immediately thereafter substantially neutralizing said wool fibers.

Enzymatic Unhairing and Dewooling Process. U. S. Pat. 2,857,316. Otto Grimm, Darmstadt, Germany, assignor to Rohm & Haas G. m. b. H., Darmstadt, Germany. Appl. Sept. 8, 1955. Claims priority, appl. Germany March 30, 1955.—1. A method of unhairing skins and hides which comprises applying proteolytic enzyme, in powder form, to a hide, permitting enzymatic action to take place at ambient temperature without immersion in water, and thereafter unhairing the treated hide.

Treatment of Skins and Hides. U. S. Pat. 2,857,317. Otto Grimm, Darmstadt, Germany, assignor to Rohm & Haas G. m. b. H., Darmstadt, Germany. Appl. Oct. 22, 1956. Claims priority, appl. Germany March 30, 1955.—1. A method of preparing a hide for tanning which comprises applying proteolytic enzyme, in powder form, to a hide, permitting enzymatic action to take place at ambient temperature without immersion in water, unhairing the treated hide, and bating the unhaired hide enzymatically in an acid bating liquor having a pH of from 3 to 5.

Fur-glazing Composition and Method for Preparing Same. U. S. Pat. 2,861,949. Willis C. Ware, Chicago, Ill. Appl. Sept. 10, 1956.—1. A fur-glazing

composition suitable for application by spraying and comprising water having dispersed therein a dialkyl dimethyl ammonium salt wherein each alkyl group contains from 8 to 18 carbon atoms, a silicone oil selected from the group consisting of polydimethyl siloxane and polydiethyl siloxane and a material selected from the group consisting of a polyethylene glycol ether of alkyl phenol being a condensation product of one molecule of an alkyl phenol wherein the alkyl group contains from 8 to 10 carbon atoms with from 2 to 10 molecules of ethylene oxide and a tertiary amine having the following structural formula:



wherein R signifies an alkyl group containing from 8 to 18 carbon atoms and wherein x and y are numbers totalling from 2 to 10.

BOOK REVIEWS

Taschenbuch für die Wachsindustrie (Pocketbook for the Wax Industry). C. Lüdecke and L. Ivanovszky. 4th ed. Stuttgart: Wissenschaftliche Verlagsgesellschaft M.B.H., 1958. 718 pp.; cloth, small 8vo. 48 DM.—The preceding edition of this work (1948) apparently was not reviewed in this journal, although two books in the same field by Ivanovszky have been noticed (*JALCA*, **48**, 54 [1953]; **49**, 694 [1954]). The scope of the book will be apparent from its Table of Contents, which reads as follows: Part I, Introduction; Part II, Natural Waxes; Part III, Mineral Waxes; Part IV, Chemically Altered and Synthetic Waxes; Part V, Commercial Varieties, Properties, and Evaluation of Waxes; Part VI, Auxiliary Materials for the Wax Industry; Part VII, Uses of Waxes; Part VIII, Examination of Waxes and Wax Products; Part IX, Tables. Part VII includes a 40-page discussion of shoe creams, and Part VIII includes 10 pages devoted to the analysis of such polishes. H.B.M.

The Chemistry and Technology of Leather. Vol. 2. Types of Tannages, ed. F. O'Flaherty, W. T. Roddy, and R. M. Lollar. A.C.S. Monograph Series No. 134. New York: Reinhold Publishing Corporation, 1958. 554 pp. $16.50.—This volume contains sixteen chapters written by sixteen chemists with one author contributing two chapters and one chapter coauthored by two writers. It has an international flavor with five foreign contributors. The fact that it is possible to restrict this volume to a single facet of leather manufacture makes it somewhat more coherent than the first volume. It is easier to read and digest.

The inclusion of two chapters on the definition of tanning, one on criteria and one on shrinkage phenomena, shows the inadequacy of the classical statement that tanning is the conversion of a putrescible protein to a nonputrescible protein. This reviewer is thoroughly in accord with Lollar's statement that a "tannage must convert skin into a fabric which can be finished into a com-

mercially useful material called leather", which is reinforced later by Kay's statement that leather "must have imparted to it many other properties [than nonputrescibility] to make it suitable for the particular end use to which it is to be put." Possibly these clear descriptions will discourage the present casual use of the term "tanning agent" to cover everything from low-molecular-weight phenols to impregnating and waterproofing agents.

Nayudamma points out that the shrinkage of collagen and leather is a molecular phenomenon analogous to melting and that it is a rate process. The latter is particularly important from a practical standpoint, since there must be a wide margin between the shrinkage temperature following tanning and the temperatures of subsequent wet operations, such as fatliquoring and drying, to avoid loss of measurement and damage to the leather.

The chapter on aldehyde tanning by Mellon is noteworthy for its thorough coverage of the literature of protein chemistry and its systematic discussion of the reaction of formaldehyde with different groups in the amino acids.

The remarkable progress made in the last ten years on the chemistry of vegetable tannins by the application of relatively new techniques is ably presented by White. If fundamental research can be continued, further clarification of the structures of natural organic tannins can be confidently anticipated. The interesting search for new industrial uses for vegetable tannins is caused by the decrease in the production of sole leather. The extract manufacturers undoubtedly regret that this research was not started in earnest many years ago.

Kay's chapter on the process of vegetable tannage is particularly useful for its description of the properties produced in leather by the individual vegetable tannins. These are surprisingly difficult to find in earlier literature and are not adequately understood by many light-leather tanners. The section on sole leather indicates that the much-maligned sole leather branch of the industry *is* doing something to lower costs, speed processing, improve quality, and diversify production, with a substantial assist from chemists.

Lollar, the author of the chapter on the mechanism of vegetable tanning, accepts the general belief that hydrogen bonding is responsible for the fixation of natural organic tannins by collagen. The restriction of this mechanism to condensed tannins by the statement that "hydrolyzable tannins . . . . . are not typical tannins" strikes this reviewer as incorrect. Chestnut, like quebracho and wattle, is characterized by a large number of phenolic hydroxyl groups and can tan equally well by hydrogen bonding, regardless of the presence of other groups. To dismiss a tanning material such as chestnut as not being typical because it is hydrolyzable is a new concept! Much more credit should be given in the literature to the pioneering work of Holmes and Cheshire which enabled the English to make satisfactory sole leather with wattle alone, and the French with chestnut alone, during World War II.

The chapters on chrome tanning, its chemistry, practice, and theory by T. C. Thorstensen, E. B. Thorstensen, and Shuttleworth respectively, provide a useful background for this all-important and most successful method of tanning. While it is generally agreed that coordinate-covalent linkages with the carboxyl groups of collagen are necessary to account for the remarkable hydrothermal stability of chrome-tanned leather, chemists still seem to be reluctant to write any formulae to picture these linkages. In contrast, numerous olated structures for the chrome complexes in water solution have been postulated for many

years; but this is not tanning. The isolation and identification of amino acid residues cross-linked with a tanning agent would be very desirable.

Somerville, the discoverer of the zirconium tannage, has written a lucid and learned chapter about this newest mineral tannage. The fact that basic zirconium sulfate, like basic chromium sulfate, is the best salt for tanning makes the practical differences between these two tannages all the more intriguing. Generous credit has been given to French chemists for their research on this tannage, not only by numerous references but also by the inclusion of a section on the theoretical aspects largely contributed by Professor Paul Chambard and Mlle. R. Lasserre of the French Tanning School, Lyons, France.

Professor Chambard's definitive chapter on the miscellaneous tannages of alum, iron, silica, and polyphosphates provides a thorough discussion and literature survey. The theoretical aspects of the iron and silica tannages have been advanced by the rather voluminous amount of work, but this has not led to any corresponding commercial success because of the inherent weaknesses of these tannages.

The chapter on replacement synthetic tans by Turley is the most complete and revealing treatment available on this subject in book form. Formulae of typical synthetic tans which would have made the chapter even more useful were omitted by the publisher. The summary points out the large gaps still existing in our knowledge of this subject.

The chapter on resin and polymeric tannages by Chen is a generous but rather uncritical review of the subject with little paraphrasing of the literature.

A lengthy and interesting discussion of oil tanning by Küntzel makes it apparent that a substantial amount of basic research has been done in Europe on a subject that has received no attention in the United States. Of seventy references not one appeared in an American journal. In connection with his section on sulfonyl chloride tanning, the Reed process, on which the "Immergans" are based, is mentioned, but it is not clear that this is an American discovery owned by duPont. The U. S. patent numbers are not given. Since patents are the only published literature available to many industrial chemists, this reviewer feels that they should be included in a bibliography.

It is refreshing to come upon a largely practical paper after rather heavy going through theoretical sections. Butz gives the most complete review of the history and development of army retan leather that this reviewer has seen. A number of significant statements on the advantages of vegetable retanning are obviously the result of tannery experience. One error occurs on page 480. Chestnut is used to produce firm leather and should not be classed with sumac and gambier as giving fairly soft leather. Possibly the reference should have been to "sweetened" chestnut used on chrome-tanned leather having a pH above four.

The exacting service requirements of leather are well documented in the chapter by Seligsberger and Mann on the stability of tannage. The determination of the changes in the properties of leather under different conditions and over a period of time requires elaborate and expensive equipment. This expenditure by the Army is amply justified by significant results. The effects of perspiration, microorganisms, and enzymes on leather are reviewed.

A number of typographical and other errors escaped correction. On both the outside and inside of the dust wrapper "tannery" should read "tanning". On p. XIV, under 29, "retainning" should be "retanning". On p. 3 the hyphen is omitted from the chemical names, whereas it is inserted correctly on p. 9. On p. 23, line 10, "bounded" should be "bonded". On p. 25 the first line of the summary should read: "It has been indicated that the useful properties we recognize in leather are the result of a combination of chemical and mechanical operations employed in leather manufacture." On p. 186, in Figure 19-3, the bottom line of numbers is reversed; 13 should follow 12, and 24 should be under 1. On p. 213 "phenomenen" should be "phenomenon". On p. 243 "stearic" should read "steric". On p. 391 the meaning is not clear where the words "phenol cresol" occur together. With reference to the plural of nucleus in Chapter 26 this reviewer will continue to use *nuclei*, rather than the awkward new word *nucleuses*, regardless of dictionary sanction. On p. 471, near the middle of the page, "adoptable" should be "adaptable", and on p. 487 it is believed that "adapted" should be "adopted". On p. 477 the correct name of "J. R. Evans Tanning Company" is "John R. Evans and Company." On p. 504, line 19, "potassium" standing by itself implies metallic potassium, whereas a salt of potassium is meant. No attempt was made to spot-check references, but the one to vanadium in the index does not occur on p. 100.

In a book of this nature it is inevitable that the approach to a particular subject reflects the point of view of the author. Not only is this true of the theoretical aspects, where agreement is not universal, but even in practice tanners will argue endlessly about the best procedures for making leather. However, this is not unique in leather chemistry and makes for progress. The amount of effort expended in writing these chapters and in assembling the book is impressive. Besides summarizing our present knowledge this volume serves as a useful guide to the literature. While reading the book this reviewer marked a number of important references which must be read for the first time in the near future.

W. WINDUS

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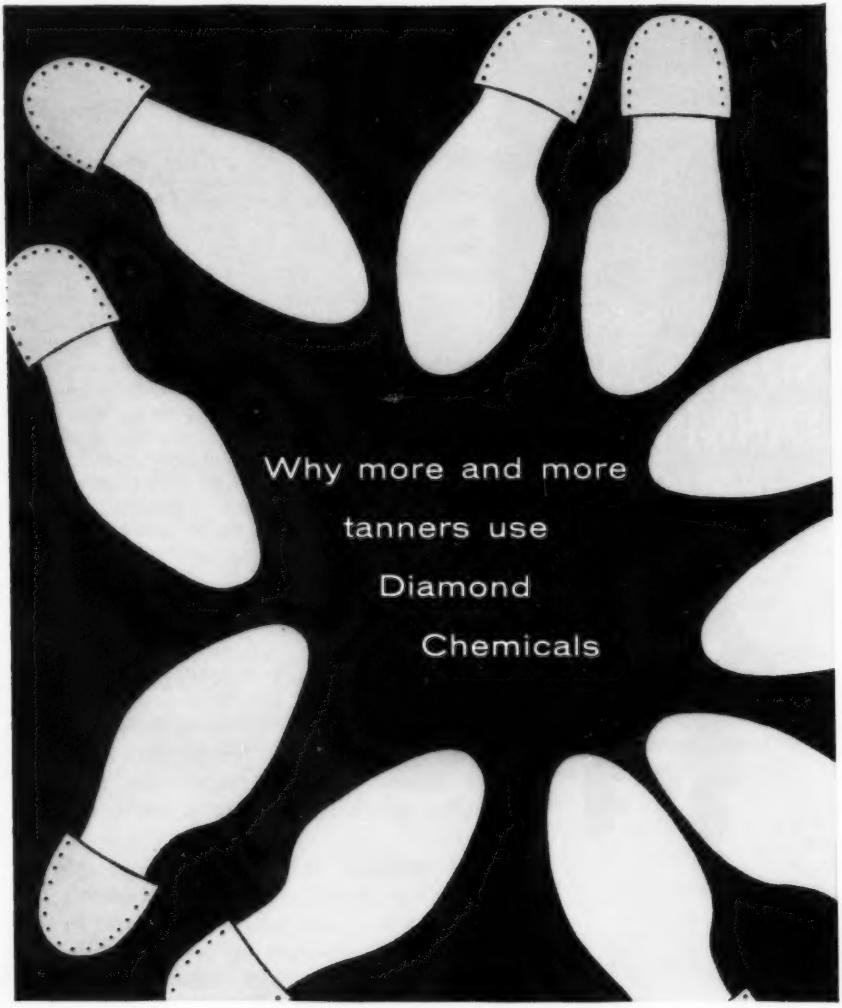

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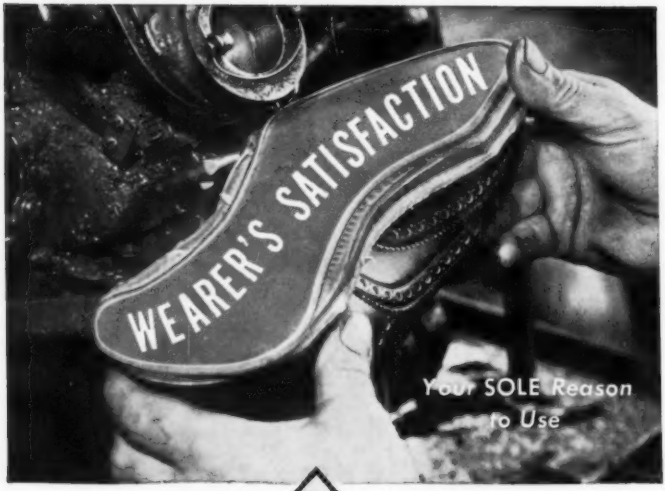

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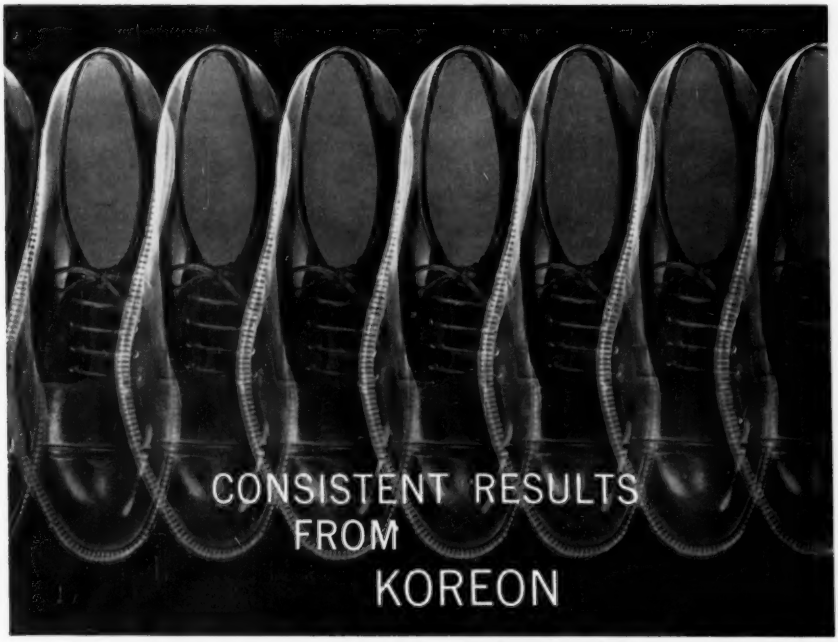

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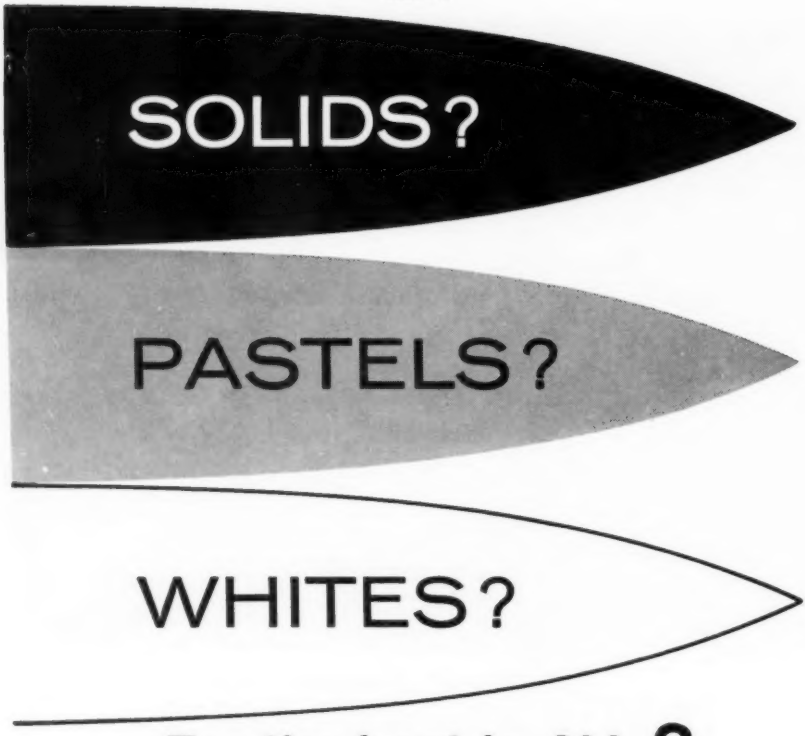

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